

## REMARKS

As background to the present request for reconsideration, following several Official Actions, the Applicant conducted an Interview with the present Examiner and Supervisory Examiner Carlson on June 24, 2008. As reflected in the Interview Summary (copy attached), it was agreed that Applicant would amend the application with regard to the effective range of the cleansing agent in the claimed composition, and would provide support from the specification for that range. During the Interview, it was acknowledged that Applicants' original disclosure included a reference to albumin being dissolved in a "conventional liquid soap base", and that Applicant would provide information showing reflecting what amount of the cleansing agent was reflected by the reference to a conventional liquid soap base. In an amendment filed July 11, 2008 as a result of the discussion during the Interview (copy attached), Applicant provided documentary evidence showing that a "conventional liquid soap base" contained 5 to 55% of the cleansing agent. This documentary evidence was presented in the form of a 27-page Appendix, including three US patents and an additional printout evidencing the percentage of the cleansing agent in liquid soap. The documentary evidence clearly showed that the range of cleansing agent in the claims was supported by the original disclosure and the reference to a "conventional liquid soap base."

In response to Applicant's submission, and despite the fact that this very issue had been discussed in the Interview between Applicant and the Examiner and supervisory Examiner Carlson, the Examiner issued a rejection dated October 16, 2008 which reflected that the Examiner had totally failed to consider Applicant's voluminous

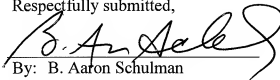
evidence regarding the support for the introduction of a range of the cleansing agent into the claims. In particular, although the Examiner acknowledged that the amendments would overcome virtually all of the prior art rejections, the Examiner objected to the amendments to the claims on the basis of new matter. In the rejection at Page 3 of the Official Action, the Examiner completely failed to address Applicant's showing regarding the established percentage of cleansing agent in a "conventional liquid soap base", and instead merely indicated that Applicant had to "point out specification support for the phrase in the specification as originally filed."

However, the support for the amendments to the claims was provided in the voluminous evidence provided with Applicant's submission of July 11, 2008. To the extent the Examiner ignored this evidence because he was merely looking for "the phrase in the specification as originally filed", such an approach in completely contradicted by the law. Indeed, it is black letter law that the specification does **not** need to provide support for an amendment *in haec verba*, but instead the claims are supported wherever a disclosure is made in the specification either expressly or inherently. See *All Dental Prodx, LLC v. Advantage Dental Prods., Inc.*, 309 F.3d 774 (Fed. Cir. 2002). As the Federal Circuit recognized, the specification need not describe the claimed subject matter in exactly the same terms as used in the claims when one skilled in the art would recognize upon reading the specification that the new language reflects what the specification shows has been invented.

In this case, the Applicants expressly disclosed use of albumin in a conventional liquid soap base, and as evidenced in Applicant's response filed July 11, 2008, one skilled in the art would have recognized what range of cleansing agent was referred to by this disclosure. Accordingly, Applicant's amendments were completely supported by the specification and did not constitute new matter.

Following the complete lack of consideration of Applicant's previous evidence of support for its claim amendments, Applicant has tried repeatedly through numerous communications with the Examiner and multiple Supervisors to have the previous action reconsidered, and to get the Examiner to review the material in support of the claim amendments which should have been done originally. However, in light of the Patent Office's failure to rectify this situation despite these many communications, Applicant's has been forced to submit this request for reconsideration which hopefully will get the Examiner to do what he should have done in response to Applicant's submission of July 11, 2008, namely acknowledge that one skilled in the art would have recognized that the ranges in the claims as amended are supported in the original disclosure. Applicant submits that this should result in the removal of the new matter rejection and an indication of the allowability of all or substantially all of the present claims.

Respectfully submitted,



By: B. Aaron Schulman

Registration No.: 31,877

Date: May 26, 2009

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
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09/740,821

12/21/2000

Daniel C. Carter

P06652US01/BAS

6567

881 7590 06/30/2008

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EXAMINER

LIU, SAMUEL W

ART UNIT

PAPER NUMBER

1656

MAIL DATE

DELIVERY MODE

06/30/2008

PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

COPY

<p align="center"><b>Interview Summary</b></p>	<b>Application No.</b> 09/740,821	<b>Applicant(s)</b> CARTER, DANIEL C.	
	<b>Examiner</b> SAMUEL W. LIU	<b>Art Unit</b> 1656	

All participants (applicant, applicant's representative, PTO personnel):

(1) Samula Liu.

(3) Bill Schulman.

(2) Karen C. Carlson.

(4) Daniel C. Carter.

Date of Interview: 24 June 2008.

Type: a) ☐ Telephonic b) ☐ Video Conference

c) ☒ Personal [copy given to: 1) ☐ applicant 2) ☐ applicant's representative]

Exhibit shown or demonstration conducted: d) ☐ Yes e) ☒ No.

If Yes, brief description: \_\_\_\_\_.

Claim(s) discussed: 50 and 54.

Identification of prior art discussed: US Pat. Nos. 5254331 and 5641483.

Agreement with respect to the claims f) ☐ was reached. g) ☐ was not reached. h) ☒ N/A.

Substance of Interview including description of the general nature of what was agreed to if an agreement was reached, or any other comments: See Continuation Sheet.

(A fuller description, if necessary, and a copy of the amendments which the examiner agreed would render the claims allowable, if available, must be attached. Also, where no copy of the amendments that would render the claims allowable is available, a summary thereof must be attached.)

THE FORMAL WRITTEN REPLY TO THE LAST OFFICE ACTION MUST INCLUDE THE SUBSTANCE OF THE INTERVIEW. (See MPEP Section 713.04). If a reply to the last Office action has already been filed, APPLICANT IS GIVEN A NON-EXTENDABLE PERIOD OF THE LONGER OF ONE MONTH OR THIRTY DAYS FROM THIS INTERVIEW DATE, OR THE MAILING DATE OF THIS INTERVIEW SUMMARY FORM, WHICHEVER IS LATER, TO FILE A STATEMENT OF THE SUBSTANCE OF THE INTERVIEW. See Summary of Record of Interview requirements on reverse side or on attached sheet.

/Karen Cochrane Carlson, Ph.D./  
 Primary Examiner, Art Unit 1656  
 Examiner's signature, if required

Examiner Note: You must sign this form unless it is an  
 Attachment to a signed Office action.

**TRANSMITTAL FORM**

(for all correspondence after initial filing)

Application # 09/740,821  
 Confirmation # 6567  
 Filing Date December 21, 2000  
 First Inventor CARTER  
 Art Unit 1653  
 Examiner Liu, Samuel W.  
 Docket # P06652US01/BAS

Total number of pages in this submission =

**ENCLOSURES** (check all that apply)

- ☒ Fees calculated below  
☒ Amendment/Reply  
☒ including Attachment(s)  
☐ After Final Amendment/Reply  
☐ including Attachment(s)  
☒ Extension of Time Petition  
☒ Appendix

- ☐ Reply to Missing Parts/Incomplete Application  
☐ Certified Copy of Priority Document(s)  
☐ Information Disclosure Statement  
☐ Drawing(s)  
☐ Terminal Disclaimer  
☐

**FEES CALCULATION:** For claims if required and/or other fees as shown below:

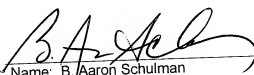
FEES CALCULATION: For claims if required and/or other fees as shown below:					\$
	NOW	Previously Paid For	Present Extra	Rate	
<input checked="" type="checkbox"/> TOTAL CLAIMS	8	- 20	0	X \$ 50 =	0
<input checked="" type="checkbox"/> INDEPENDENT CLAIMS	4	- 5	0	X \$ 210 =	0
TOTAL OF ABOVE CLAIMS FEES =					0
<input type="checkbox"/> Reduction by 1/2 for <b>small entity status</b> of applicant					0
SUBTOTAL =					525
<input checked="" type="checkbox"/> Fee for extension of time (per attached Petition)					
<input type="checkbox"/> Other fee for					
TOTAL OF ALL FEES =					525

☒ Payment of \$ 525.00 is made by:

- ☒ CREDIT CARD PAYMENT FORM - PTO-2038 submitted concurrently herewith.  
☐ ELECTRONIC FUNDS TRANSFER - submitted concurrently herewith.

- ☒ The Director is authorized to charge any fee, additional fee or extension fee due in connection herewith to Deposit Account No. 12-0555:  
 (1) If no payment or an insufficient payment is enclosed and a fee is due in connection herewith; or  
 (2) If no petition for extension of time is enclosed but an EOT is required - and in this event, applicant hereby petitions under 37 CFR 1.136(a) for an extension of time of as many months as are required to render this submission timely.

Date: July 11, 2008

Signed By  Name: B. Aaron Schulman  
 Attorney of Record Registration No.: 31,877

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<b>PETITION FOR EXTENSION OF TIME UNDER 37 CFR 1.136(a) FY 2008</b>	Application #	09/740,821
	Confirmation #	6567
	Filing Date	December 21, 2000
	First Inventor	CARTER
	Art Unit	1653
	Examiner	Liu, Samuel W.
	Docket #	P06652US01/BAS

This is a request under the provisions of 37 CFR 1.136(a) to extend the period for filing a reply in the above identified application.

The requested extension and fee under 37 CFR 1.17(a) are as follows.

☒ NOTE: Applicant claims small entity status.

No extension of time previously obtained.

Time Period	Per Rule	Large Entity Fee	Small Entity Fee
<input type="checkbox"/> One month	1.17(a)(1)	<input type="checkbox"/> \$ 100	<input type="checkbox"/> \$ 60
<input type="checkbox"/> Two months	1.17(a)(2)	<input type="checkbox"/> \$ 460	<input type="checkbox"/> \$ 230
<input checked="" type="checkbox"/> Three months	1.17(a)(3)	<input type="checkbox"/> \$ 1050	<input checked="" type="checkbox"/> \$ 525
<input type="checkbox"/> Four months	1.17(a)(4)	<input type="checkbox"/> \$ 1640	<input type="checkbox"/> \$ 820
<input type="checkbox"/> Five months	1.17(a)(5)	<input type="checkbox"/> \$ 2230	<input type="checkbox"/> \$ 1115

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- (1) if no payment or an insufficient payment is enclosed and a fee is due in connection herewith; or
- (2) if a further fee and petition for extension of time are required at this time - and in this event, applicant hereby petitions under 37 CFR 1.136(a) for any further extension of time of as many months as are required to render this submission timely.

Date: July 11, 2008

Signed By   
Attorney of Record Name: B. Aaron Schulman  
Registration No.: 31,877

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<input type="checkbox"/> <b>PATENT MAINTENANCE FEE:</b> Patent No.:          Appln. No.:		
<input type="checkbox"/> <b>TRADEMARK FEE - For:</b> Appln. No.:          Regis. No.:          Mark:		
Attorney Docket No.: P06652US01/BAS		

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<b>AMENDMENT</b>	Application #	09/740,821
	Confirmation #	6567
	Filing Date	December 21, 2000
	First Inventor	CARTER
	Art Unit	1653
	Examiner	Liu, Samuel W.
	Docket #	P06652US01/BAS

Commissioner for Patents  
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Alexandria, VA 22313-1450

S I R:

In response to the Official Action dated January 11, 2008, please amend the above identified application as follows.

**Amendments to the Claims** are reflected in the listing of the claims provided herewith in **Attachment A**.

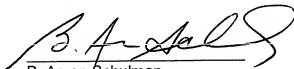
**Remarks to this Amendment** are provided herewith in **Attachment B**.

An **Appendix** with references relating to liquid soaps is also attached hereto.

In light of the amendments made and the remarks provided herewith, this case has been placed in condition for immediate allowance.

Respectfully submitted,

STITES & HARBISON PLLC



B. Aaron Schulman  
Registration No. 31877

July 11, 2008

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## ATTACHMENT A

### Amendment to the Claims

*This listing of claims will replace all prior versions, and listings, of claims in the application:*

1-49 (Canceled).

50. (Currently amended) A hypoallergenic cleansing composition for skin or hair consisting essentially of recombinant human serum albumin and a cleansing agent, vehicle, carrier or excipient, wherein the human serum albumin is present in an amount sufficient to be absorbed into skin or hair, wherein the cleansing agent, vehicle, carrier or excipient is present in an amount effective to cleanse skin or hair, and wherein the cleansing agent, vehicle, carrier or excipient is dissolved in a liquid soap, and wherein the cleansing agent, vehicle, carrier or excipient is present in the range of about 5 to 55% by weight of the liquid soap.

51. (Canceled).

52. (Currently amended) The cleansing composition according to claim 50 wherein the serum albumin comprises 1 to 4060 mg/ml of the liquid soap.

53. (Previously presented) The cleansing composition according to claim 50 wherein the serum albumin is present at a concentration of 40 mg/ml.

54. (Currently amended) A hypoallergenic cleansing composition for skin or hair comprising recombinant human serum albumin and a cleansing agent, vehicle, carrier or excipient in amounts effective to be absorbed into skin or hair and to cleanse skin or hair wherein the serum albumin is dissolved in liquid soap, and wherein the cleansing agent, vehicle, carrier or excipient is present in an amount of about 5 to 55% by weight of the liquid soap.

55. (Currently amended) The cleansing composition according to claim 54 wherein the serum albumin comprises 1 to 4060 mg/ml of the liquid soap.

Attachment A

56. (Previously presented) The cleansing composition according to claim 54 wherein the serum albumin is present at a concentration of 40 mg/ml.

57. (Currently amended) A hypoallergenic cleansing composition for skin or hair consisting essentially of recombinant human serum albumin and a liquid soap wherein the human serum albumin is present in an amount sufficient to be absorbed into skin or hair, and the liquid soap contains a cleansing agent, vehicle, carrier or excipient that is present in an amount of about 5 to 55% by weight of the liquid soap~~is present in an amount effective to cleanse skin or hair.~~

58. (New) A hypoallergenic cleansing composition for skin or hair consisting essentially of human serum albumin and a cleansing agent, vehicle, carrier or excipient, wherein the human serum albumin is present in an amount sufficient to be absorbed into skin or hair, wherein the ratio of human serum albumin to the cleansing agent, vehicle, carrier or excipient is about 0.3:1 to 2.4:1 by weight, and wherein the cleansing agent, vehicle, carrier or excipient is dissolved in a liquid soap.

COPY

industry to mean about 5 to 55% of the cleansing agent (e.g., sodium laurel sulfate or ammonium laurel sulphate) in the liquid soap composition. Applicant has provided herewith an Appendix of various patents and other materials showing that generally a conventional liquid soap will contain roughly 5 to 55% of the cleansing agent, and this range has now been incorporated into the present claims. As also indicated in page 11, the amount of human serum albumin used in the composition may be in the range of 1 to 60 mg/ml (and Claims 52 and 55 have been amended accordingly), and thus this translates to a ratio of serum albumin to cleansing agent of from about 0.3:1 to about 2.4:1, as now reflected in new Claim 58. As indicated below, it is clear that none of these compositions are disclosed or made obvious in any prior art reference, and that the claims now distinguish over those references.

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In the Official Action, the Examiner made a minor objection to Claim 50 with regard to the term "cleansing agent, vehicle, carrier or excipient", and Applicant submits that the terms "vehicle, carrier or excipient" refer to the cleansing agent and are merely different forms of this agent. The language to Claim 50 has been amended to make this clear, and the Examiner's rejection under Section 112 is respectfully traversed.

In the Official Action, the Examiner rejected Claims 50 and 52-57 on the basis of Mausner U.S. patent 5,254,331 which relates to a skin cream composition and not to a cleansing composition or a liquid soap. As was discussed during the interview between Applicant and the Examiner, the Mausner composition does not disclose or make obvious Applicant's claimed invention because it relates to a skin cream, and the extremely minor amount of an agent that in other compositions might be used for cleansing (e.g., steareth-21) would not in any event be an effective amount in that

composition for that purpose. However, even further, Applicant has now quantified the amount of the cleansing agent in the claims in the manner suggested by the Examiner, and this amount clearly distinguishes the claims from the Mausner patent which is not a cleansing composition, and which does not in any event disclose or approach the amount of the cleansing agent as utilized in Applicant's claims. Accordingly the Examiner's rejection on the basis of Mausner is respectfully traversed and should be withdrawn.

Finally, in the Official Action, the Examiner had a similar rejection of claims 50, 52, 54-55 and 57 on the basis of Beaulieu US Patent 5,641,483 which once again is not a cleansing composition but which is a wound healing formulation. Apparently, the reference was only cited because of the inclusion of a very small amount of "cetech-20", although this amount was not an effective amount in that composition for purposes of cleansing. Once again, as was discussed during the interview between Applicant and the Examiner, the Beaulieu composition does not disclose or make obvious Applicant's claimed invention because it relates to a wound healing formulation, and the extremely minor amount of an agent that in other compositions might be used for cleansing (e.g., cetech-20) would not in any event be an effective amount for that purpose. However, even further, Applicant has now quantified the amount of the cleansing agent in the claims in the manner suggested by the Examiner, and this amount clearly distinguishes the claims from the Beaulieu patent which is not a cleansing composition, and which does not in any event disclose or approach the amount of the cleansing agent as utilized in Applicant's claims. Accordingly the Examiner's rejection on the basis of Beaulieu is respectfully traversed and should be withdrawn.

Accordingly, the Examiner's rejections on the basis of the cited prior art, insofar as applied to the claims as amended, are respectfully traversed and should be withdrawn.

In light of the amendments and arguments as set forth above, Applicants submit that the present application overcomes all prior rejections and has been placed in condition for allowance. Such action is respectfully requested.

**END OF REMARKS**

COPY

APPENDIX FOR U.S. SER. NO. 09/740,821

REFERENCES 1-4 RELATING TO LIQUID SOAPS

COPY



US005158699A

**United States Patent** [19][11] **Patent Number:** 5,158,699**MacGilp et al.**[45] **Date of Patent:** \* Oct. 27, 1992

[54] **LIQUID SOAP PERSONAL CLEANSER  
WITH CRITICAL HEAT CYCLE  
STABILIZING SYSTEM**

[75] **Inventors:** Neil A. MacGilp; Kathleen G. Baier;  
Richard M. Girardot; Efrain Torres,  
all of Cincinnati, Ohio

[73] **Assignee:** The Procter & Gamble Company,  
Cincinnati, Ohio

[\*] **Notice:** The portion of the term of this patent  
subsequent to Sep. 15, 2009 has been  
disclaimed.

[21] **Appl. No.:** 665,621

[22] **Filed:** Mar. 5, 1991

[51] **Int. Cl.<sup>5</sup>** ..... C11D 9/04; C11D 9/10;  
C11D 9/48; C11D 17/08

[52] **U.S. Cl.** ..... 252/132; 252/108;  
252/133; 252/173; 252/DIG. 5; 252/DIG. 14;  
252/142

[58] **Field of Search** ..... 252/108, 370, 173, DIG. 14,  
252/DIG. 5, 133, 132, 142

[56] **References Cited**

**U.S. PATENT DOCUMENTS**

4,190,549	2/1980	Inamura et al.	252/91
4,338,211	7/1982	Stiros	252/142
4,387,040	6/1983	Straw	252/368
4,673,525	6/1987	Small et al.	252/132
4,861,507	8/1989	Gervasio	252/108
4,917,823	4/1990	Maille, Jr.	252/548

**FOREIGN PATENT DOCUMENTS**

1235292 6/1991 United Kingdom .

**OTHER PUBLICATIONS**

Davidson et al; Soap Manufacture; vol. 1; 1953 p. 305.

*Primary Examiner*—Paul Lieberman  
*Assistant Examiner*—Erin M. Higgins  
*Attorney, Agent, or Firm*—Leonard Williamson; Thomas  
H. O'Flaherty

[57]

**ABSTRACT**

The present invention relates to a stable dispersoidal  
liquid soap cleansing composition comprising:

A. from about 5% to about 20% by weight of potassium  
fatty acid soap;

B. from about 2.5% to about 18% C<sub>8</sub>-C<sub>22</sub> free fatty  
acid; wherein said fatty acid has an Iodine Value of  
from zero to about 15; and a titer of from about 44 to  
about 70;

C. from about 55% to about 90% water; and po0 D.  
from about 0.1% to about 4% of a stabilizer selected  
from the group consisting of: from about 0.1% to  
about 2.0% of an electrolyte; and from 0% to about  
2.0% of a polymeric thickener; and mixtures thereof;  
and

wherein said soap and said free fatty acid have a weight  
ratio of about 1:0.5 to about 1:1; and wherein said liquid  
has an initial viscosity of from about 4,000 cps to about  
100,000 cps and a cycle viscosity of from about 10,000  
cps to about 100,000 cps.

12 Claims, No Drawings

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# LIQUID SOAP PERSONAL CLEANSER WITH CRITICAL HEAT CYCLE STABILIZING SYSTEM

## TECHNICAL FIELD

The present invention is related to liquid soap products, especially pumpable facial cleansers and bath/shower compositions which are formulated for viscosity control or phase stability.

## BACKGROUND ART

Liquid personal cleansing compositions are well known. patents disclosing such compositions are U.S. Pat. Nos.: 3,697,644, Laiderman, issued Oct. 10, 1972; 3,932,610, Rudy et al., issued Jan. 13, 1976; 4,031,306, DeMartino et al., issued Jun. 21, 1977; 4,061,602, Oberstar et al., issued Dec. 6, 1977; 4,387,040, Straw, issued Jun. 7, 1983; and 4,917,823, Maile, Jr., issued Apr. 17, 1990; and Brit. Pat. No. 1,235,292, published Jun. 9, 1971.

Most liquid soaps comprise mostly "soluble," "unsaturated," shorter chains, e.g., lauric/oleic soaps for phase stability. This, however, compromises lather quality or mildness.

Brit. Pat. 1,235,292, supra, discloses a mix of K/Na soap; at least 5% K soap; and 0.1-5% alkyl cellulose. The '292 soaps are natural. Natural fatty acids contain some unsaturation and therefore have higher Iodine Values and lower titers. The '292 exemplified liquid soaps contain from about 17% to about 21.5% soap and up to 1% free fatty acid.

U.S. Pat. No. 4,387,040, supra, discloses a stable liquid K soap containing a viscosity controlling agent composed of coco-DEA and sodium sulfate. Saturated acid soaps of C<sub>12</sub>-C<sub>14</sub> are used. The viscosity of the '040 soap is 1,000-1,500 cps at 25° C. RVT/Spindle 3/10 rpm. Free fatty acid is not taught. Some of the '040 formulations contain electrolyte and polymeric thickener, but those formulations are disclosed as unstable. It should also be noted that lauric acid soap is a relatively harsh soap and when used at higher levels (as used in '040) works against product mildness.

Newtonian liquids which are too viscous are more difficult to pump than shear thinning liquids. Liquid "soap" products on the market today are mostly Newtonian or only slightly to moderately shear thinning liquids.

While it is known to use natural potassium (K) soap to make liquid cleansing compositions, there is no teaching or suggestion of solutions to certain problems encountered with superfatated, saturated, low Iodine Value (IV), higher fatty acid (FFA) soaps.

Specifically, phase stability, good lather, and viscosity control and stability are heretofore unsolved, or only partially solved, problems in this art.

While these previously disclosed liquid soap formulations are not subject, or are subject to a lesser degree, to one or more of the above-described deficiencies, it has been found that further improvements in physical stability and stability against rheological properties variations with time or temperature are desired to increase the shelf life of the product and thereby enhance consumer acceptance.

It is, therefore, an object of the present invention to provide a liquid cleansing bath/shower soap composition which is phase stable, shelf stable, lathers well, and is cosmetically attractive.

It is a further object of the present invention to provide a liquid soap cleansing composition which is relatively mild.

It is a still further object of the present invention to provide a viscous, high shear thinning liquid soap cleansing composition which is pumpable from a standard hand pressure pump container.

These and other objects of the present invention will become obvious from the detailed description which follows.

## SUMMARY OF THE INVENTION

The present invention relates to a stable dispersoidal liquid soap cleansing composition comprising:

- A. from about 5% to about 20% by weight of potassium fatty acid soap;
- B. from about 2.5% to about 18% C<sub>8</sub>-C<sub>22</sub> free fatty acid; wherein said fatty acid has an Iodine Value of from zero to about 15; and a titer of from about 44 to about 70;
- C. from about 55% to about 90% water; and
- D. from about 0.1% to about 4% of a stabilizer selected from the group consisting of: from about 0.1% to about 2.0% of an electrolyte; and from 0% to about 2.0% of a polymeric thickener; and mixtures thereof; and wherein said soap and said free fatty acid have a weight ratio of about 1:0.5 to about 1:1; and wherein said liquid has an initial viscosity of from about 4,000 cps to about 100,000 cps and a cycle viscosity of from about 10,000 cps to about 80,000 cps.

## DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a stable dispersoidal liquid soap cleansing composition comprising: 55% to 90%, preferably 60% to 80%, water; 5% to 18%, preferably 6% to 14%, of mostly insoluble saturated (low IV) higher fatty acid potassium soap; 3% to 18%, preferably 4% to 9%, of free fatty acids.

The liquid soap preferably contains from about 0.2% to about 5%, preferably from about 0.3% to about 3%, of a stabilizing ingredient selected from the group consisting of: polymeric thickener, electrolyte, or nonionic, and mixtures thereof; preferably from 0.1% to 2% of a thickener; 0.1% to 3% electrolyte; and 0.1% to 2% nonionic, and mixtures thereof. One or more of these ingredients improves the stability of the liquid soap. Preferably the liquid soap contains from about 0.1% to about 2% of a thickener. Preferably the liquid soap contains from about 0.1% to about 3% electrolyte. Preferably the liquid soap contains from about 0.1% to about 2% nonionic.

The soap and the free fatty acids have a ratio of above about 1:0.5 to about 1:1 and preferably from about 1:0.6 to about 1:0.8. The preferred fatty acid matter is a mixture of the following saturated fatty acids on a total fatty matter basis:

- C<sub>12</sub> at a level of about 13.5% ± 5% ± 2%;
- C<sub>14</sub> at a level of about 35.5% ± 15% ± 5%;
- C<sub>16</sub> at a level of about 24% ± 10% ± 5% ± 3%;
- C<sub>18</sub> at a level of about 29% ± 10% ± 5% ± 3%.

The fatty acid matter of the present invention has an IV of from zero to about 15, preferably below 10, more preferably below 3; and a titer of from about 44 to about 70, preferably from about 50 to 70, more preferably from about 59 to about 70.

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The liquid soap has a viscosity of 4,000 cps to 100,000 cps, preferably 10,000 cps to about 80,000 cps at about 25°. The preferred composition has a viscosity of 15,000-45,000 cps and, more preferably, a viscosity of 20,000-40,000 cps.

The liquid soap is called a dispersoid because at least some of the fatty matter at the levels used herein is insoluble. The level of water in the compositions is typically from about 55% to about 90%, preferably from about 60% to about 80%.

The chemical properties of some preferred pure saturated acids which have Iodine Values of zero are set out below in the Pure Acid Table.

Pure Acid Table				
Acid	Chain Length	Acid Value	Molecular Weight	Titer °C.
Decanoic	C-10	326	172	
Lauric	C-12	280	200	44.2
Myristic	C-14	246	228	54.4
Pentadecanoic	C-15	231	242	
Palmitic	C-16	219	256	62.9
Margaric	C-17	207	270	
Stearic	C-18	197	284	69.6
Nonadecanoic	C-19	188	298	
Arachidic	C-20	180	312	
Behenic	C-22	165	340	

The titers or I.V.'s of "natural" acids are outside of the selected fatty matter of the present invention.

Palm Kernel Acid Table			
Acid	Chain Length		Wt. %
<u>Saturated:</u>			
Oleic	C-8		3
Decanoic	C-10		3
Lauric	C-12		50
Myristic	C-14		18
Palmitic	C-16		8
Stearic	C-18		2
<u>Unsaturated:</u>			
Oleic	C-18 = 1		14
Linoleic	C-18 = 2		2
Iodine Value:	Low		14
	High		23
Saponification Value:	Low		245
	High		255
Titer, °C. (Fatty Acid):	Low		20
	High		28

Note that the titer is low.

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Coconut Acid Table			
Acid	Chain Length		Wt. %
<u>Saturated:</u>			
Oleic	C-8		7
Decanoic	C-10		6
Lauric	C-12		50
Myristic	C-14		18
Palmitic	C-16		8.5
Stearic	C-18		3
<u>Unsaturated:</u>			
Oleic	C-18 = 1		6
Linoleic	C-18 = 2		1
Linolenic	C-18 = 3		0.5
Iodine Value:	Low		7.5
	High		10.5
Saponification Value:	Low		250
	High		264
Titer, °C. (Fatty Acid):	Low		20

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Coconut Acid Table		
Acid	Chain Length	Wt. %
	High	24

The Iodine Value of coconut acid is acceptable, but its titer is low.

Tallow BFT Table		
Acid	Chain Length	Wt. %
<u>Saturated:</u>		
Myristic	C-14	3
Pentadecanoic	C-15	0.5
Palmitic	C-16	24
Margaric	C-17	1.5
Stearic	C-18	20
<u>Unsaturated:</u>		
Myristoleic	C-14 = 1	1
Palmitoleic	C-16 = 1	2.5
Oleic	C-18 = 1	43
Linoleic	C-18 = 2	4
Linolenic	C-18 = 3	0.5
Iodine Value:	Low	45
	High	50
Saponification Value:	Low	192
	High	202
Titer, °C. (Fatty Acid):	Low	40
	High	45

Another important attribute of the preferred liquid soap of the present invention is its pumpability, particularly after storage over a cycle of temperatures. A less preferred liquid product is one in which its initial viscosity is pumpable, but there is an unacceptable increase in its viscosity which makes it unpumpable after heating to a temperature of 45° C. for about 8 hours and cooling to room temperature. The more preferred liquid soaps of the present invention can withstand more than one such cycle.

The term "pumpable" as used herein means that the liquid soap can be pumped from a standard glass or plastic container having a hand pressure actuated pump on the order of a commercially available one sold by Calmar Co., Cincinnati, Oh., under the trade name of Dispenser SD 200, with a delivery of about 1.7 cc of the liquid soap. Another standard pump is sold by Specialty Packaging Products, Bridgeport, Conn., under the trade name L.P.D.-2 Pump. This pump delivers about 1.7 cc of liquid.

The "shelf viscosity" or "cycle viscosity" of a liquid soap product is defined herein as its viscosity after subjection to one or more temperature cycles. This is used to describe the shelf or storage stability of liquid soaps which are formulated for use in a standard pressure actuated pump dispenser. The preferred product is formulated to provide the desired phase stability, viscosity and lather. It does not separate or become too viscous after heating and cooling under ambient conditions.

The terms "Initial Viscosity" and "Cycle Viscosity" as used herein are defined according to the methods taught herein, unless otherwise indicated. In short, the "Cycle Viscosity" is measured after the liquid soap has gone through a cycle of 49.5° C. for 8 hrs. and returned to 25° C. The term "viscosity" as used herein means both of these viscosities as measured by a Brookfield RVTDV-II/Spindle TD at 5 rpm at 25° C., unless otherwise specified.

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The liquid soap product of the present invention has an Initial Viscosity of from about 10,000 cps to about 60,000 cps and/or a Cycle Viscosity of from about 15,000 cps to about 80,000 cps.

The liquid soap product of the present invention is shear thinning. Its high shear thinning factor allows it to be pumped from a standard hand pressure actuated pump, notwithstanding its relatively high viscosity of 10,000 cps to 60,000 cps.

The preferred liquid soap dispersoid has a high shear thinning factor as defined herein. Its viscosity is reduced by at least a factor of 1.5, preferably at least about 2, more preferably at least about 3. The "shear thinning factor" is:

$$\frac{\text{Viscosity at a shear rate of } 1 \text{ sec}^{-1}}{\text{Viscosity at a shear rate of } 10 \text{ sec}^{-1}}$$

Viscosities are measured on a Bohlin VOR Rheometer at room temperature (25° C.). Note: The following Bohlin viscosities are different from those measured on the Brookfield Viscometer.

E.g., a liquid soap (like Example 1B below) which has a Bohlin viscosity of about 38,000 cps, at a shear rate of about  $1 \text{ sec}^{-1}$  and a Bohlin viscosity of about 4,000 cps at a shear rate of about  $10 \text{ sec}^{-1}$ . The shear thinning factor for this liquid is about 38,000/4,000 or about 9.5.

The shear thinning factors for the present invention are from about 1.5 to about 25, preferably from about 2 to about 20, more preferably from about 3 to about 15.

Additional viscosity measurements obtained with the Bohlin Rheometer show some approximate shear thinning factors for some commercially available liquid cleansers and this invention and are set out below after the Examples.

Preferably the liquid soap contains from about 0.2% up to a total of about 5%, preferably from about 0.3% to about 3%, of a stabilizing ingredient selected from the group consisting of: from 0.1% to 2% of a thickener; 0.1% to 3% electrolyte; and 0.1% to 2% nonionic, and mixtures thereof. One or more of these ingredients can improve the stability of the liquid soap. The more dilute the liquid, the more of these stabilizing ingredients can be added.

#### Thickeners

The thickeners in this invention are categorized as cationic, nonionic, or anionic and are selected to provide the desired viscosities. Suitable thickeners are listed in the Glossary and Chapters 3, 4, 12 and 13 of the *Handbook of Water-Soluble Gums and Resins*, Robert L. Davidson, McGraw-Hill Book Co., New York, N.Y., 1980, incorporated by reference herein.

The liquid personal cleansing products can be thickened by using polymeric additives that hydrate, swell or molecularly associate to provide body (e.g., hydroxypropyl guar gum is used as a thickening aid in shampoo compositions).

The nonionic cellulosic thickeners include, but are not limited to, the following polymers:

1. hydroxyethyl cellulose;
2. hydroxymethyl cellulose;
3. hydroxypropyl cellulose; and
4. hydroxybutyl methyl cellulose.

The anionic cellulosic thickener includes carboxymethyl cellulose and the like.

The preferred thickener is xanthan gum having a molecular weight (M.W.) of from about

2,000,000  $\pm$  500,000. Each molecule has about 2,000 repeating units.

Another preferred thickener is acrylated steareth-20 methacrylate copolymer sold as Acrysol ICS-1 by Rohm and Haas Company.

The amount of polymeric thickener found useful in the present compositions is about 0.1% to about 2%, preferably from about 0.2% to about 1.0%.

#### Electrolyte

An additional requirement for the present compositions is that they contain a low level of electrolyte. Electrolytes include inorganic salts (e.g., potassium or sodium chloride), as well as organic salts (e.g., sodium citrate, potassium acetate). Potassium chloride is preferred. The amount of electrolyte varies with the type of surfactant system but should be present in finished product at a level of from about 0.1% to about 3%, preferably from about 0.25% to about 2.9%. In addition to the above-mentioned chloride and citrate salts, other salts include phosphates, sulfates and other halogen ion salts. The counter ions of such salts can be sodium or other monovalent cations as well as di- and trivalent cations. It is recognized that these salts may cause instability if present at greater levels.

#### Nonionic Stabilizer

Another preferred component of the present invention is a nonionic. The preferred nonionic is polyglycerol ester (PGE).

Groups of substances which are particularly suitable for use as nonionic surfactants are alkoxylated fatty alcohols or alkyl-phenols, preferably alkoxylated with ethylene oxide or mixtures of ethylene oxide or propylene oxide; polyglycol esters of fatty acids or fatty acid amides; ethylene oxide/propylene oxide block polymers; glycerol esters and polyglycerol esters; sorbitol and sorbitan esters; polyglycol esters of glycerol; ethoxylated lanolin derivatives; and alkanolamides and sucrose esters.

#### Optional Components

If present, the optional components individually generally comprise from about 0.001% to about 10% by weight of the composition.

The liquid cleansing bath/shower compositions can contain a variety of nonessential optional ingredients suitable for rendering such compositions more desirable. Such conventional optional ingredients are well known to those skilled in the art, e.g., preservatives such as benzyl alcohol, methyl paraben, propyl paraben and imidazolidinyl urea; other thickeners and viscosity modifiers such as C<sub>8</sub>-C<sub>18</sub> ethanolamide (e.g., coconut ethanolamide) and polyvinyl alcohol; skin moisturizers such as glycerine; pH adjusting agents such as citric acid, succinic acid, phosphoric acid, sodium hydroxide, etc.; suspending agents such as magnesium/aluminum silicate; perfumes; dyes; and sequestering agents such as disodium ethylenediamine tetracetate.

#### Surfactant

An important attribute of the preferred liquid soap personal cleansing product of the present invention is its rich and creamy lather.

The preferred composition also contains from about 1% to about 10%, preferably from about 2% to about 6%, of a high lathering synthetic surfactant.

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An important optional component of the present compositions is a lather boosting surfactant. The surfactant, which may be selected from any of a wide variety of anionic (nonsoap), amphoteric, zwitterionic, nonionic and, in certain instances, cationic surfactants, is present at a level of from about 1% to about 10%, preferably from about 2% to about 6% by weight of the liquid product.

The cleansing product patent literature is full of synthetic surfactant disclosures. Some preferred surfactants as well as other cleansing product ingredients are disclosed in the following references:

Pat. No.	Issue Date	Inventor(s)
4,061,602	12/1977	Obertar et al.
4,234,464	11/1980	Morshauer
4,472,297	9/1984	Bollich et al.
4,491,539	1/1985	Hoskins et al.
4,540,307	9/1985	Grollier
4,565,647	1/1986	Lleado
4,673,525	9/1987	Small et al.
4,704,224	11/1987	Saud
4,788,006	11/1988	Bollich, Jr. et al.
4,812,255	3/1989	Small et al.
4,830,447	4/1989	Medcalf et al.
4,906,459	3/1990	Cobb et al.
4,923,635	5/1990	Simon et al.
4,954,282	9/1990	Rys et al.

All of said patents are incorporated herein by reference. A preferred synthetic surfactant is shown the Examples herein. Preferred synthetic surfactant systems are selectively designed for appearance, stability, lather, cleansing and mildness.

It is noted that surfactant mildness can be measured by a skin barrier destruction test which is used to assess the irritancy potential of surfactants. In this test the surfactant, the lesser the skin barrier is destroyed. Skin barrier destruction is measured by the relative amount of radio-labeled water ( $^3\text{H}_2\text{O}$ ) which passes from the test solution through the skin epidermis into the physiological buffer contained in the diffuse chamber. This test is described by T. J. Franz in the *J. Invest. Dermatol.*, 1975, 64, pp. 190-195; and in U.S. Pat. No. 4,673,525, Small et al., issued Jun. 16, 1987, incorporated herein by reference, and which disclose a mild alkyl glyceryl ether sulfonate (AGS) surfactant based synbar comprising a "standard" alkyl glyceryl ether sulfonate mixture. Barrier destruction testing is used to select mild surfactants. Some preferred mild synthetic surfactants are disclosed in the above Small et al. patents and Rys et al.

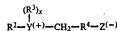
Some examples of good lather-enhancing, mild detergent surfactants are e.g., sodium lauroyl sarcosinate, alkyl glyceryl ether sulfonate, sulfonated fatty esters, and sulfonated fatty acids.

Numerous examples of other surfactants are disclosed in the patents incorporated herein by reference. They include other alkyl sulfates, anionic acyl sarcosinates, methyl acyl taurates, N-acyl glutamates, acyl isethionates, alkyl sulfosuccinates, alkyl phosphate esters, ethoxylated alkyl phosphate esters, trideceth sulfates, protein condensates, mixtures of ethoxylated alkyl sulfates and alkyl amine oxides, betaines, sultaines, and mixtures thereof. Included in the surfactants are the alkyl ether sulfates with 1 to 12 ethoxy groups, especially ammonium and sodium lauryl ether sulfates.

Alkyl chains for these surfactants are  $\text{C}_8\text{--C}_{22}$ , preferably  $\text{C}_{10}\text{--C}_{18}$ , more preferably  $\text{C}_{12}\text{--C}_{14}$ . Alkyl glycosides and methyl glucose esters are preferred mild non-

ionics which may be mixed with other mild anionic or amphoteric surfactants in the compositions of this invention. Alkyl polyglycoside detergents are useful lather enhancers. The alkyl group can vary from about 8 to about 22 and the glycoside units per molecule can vary from about 1.1 to about 5 to provide an appropriate balance between the hydrophilic and hydrophobic portions of the molecule. Combinations of  $\text{C}_8\text{--C}_{18}$ , preferably  $\text{C}_{12}\text{--C}_{16}$ , alkyl polyglycosides with average degrees of glycosidation ranging from about 1.1 to about 2.7, preferably from about 1.2 to about 2.5, are preferred.

Anionic nonsoap surfactants can be exemplified by the alkali metal salts of organic sulfuric reaction products having in their molecular structure an alkyl radical containing from 8 to 22 carbon atoms and a sulfonic acid or sulfuric acid ester radical (included in the term alkyl is the alkyl portion of higher acyl radicals). Preferred are the sodium, ammonium, potassium or triethanolamine alkyl sulfates, especially those obtained by sulfating the higher alcohols ( $\text{C}_8\text{--C}_{18}$  carbon atoms), sodium coconut oil fatty acid monoglyceride sulfates and sulfonates; sodium or potassium salts of sulfuric acid esters of the reaction product of 1 mole of a higher fatty alcohol (e.g., tallow or coconut oil alcohols) and 1 to 12 moles of ethylene oxide; sodium or potassium salts of alkyl phenol ethylene oxide ether sulfate with 1 to 10 units of ethylene oxide per molecule and in which the alkyl radicals contain from 8 to 12 carbon atoms, sodium alkyl glyceryl ether sulfates; the reaction product of fatty acids having from 10 to 22 carbon atoms esterified with isethionic acid and neutralized with sodium hydroxide; water soluble salts of condensation products of fatty acids with sarcosine; and others known in the art. Zwitterionic surfactants can be exemplified by those which can be broadly described as derivatives of aliphatic quaternary ammonium, phosphonium, and sulfonium compounds, in which the aliphatic radicals can be straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to 18 carbon atoms and one contains an anionic water-solubilizing group, e.g., carboxy, sulfonate, sulfate, phosphate, or phosphonate. A general formula for these compounds is:



wherein  $\text{R}^2$  contains an alkyl, alkenyl, or hydroxy alkyl radical of from about 8 to about 18 carbon atoms, from 0 to about 10 ethylene oxide moieties and from 0 to 1 glyceryl moiety; Y is selected from the group consisting of nitrogen, phosphorus, and sulfur atoms;  $\text{R}^3$  is an alkyl or monohydroxyalkyl group containing 1 to about 3 carbon atoms; X is 1 when Y is a sulfur atom and 2 when Y is a nitrogen or phosphorus atom;  $\text{R}^4$  is an alkylene or hydroxyalkylene of from 1 to about 4 carbon atoms and Z is a radical selected from the group consisting of carboxylate, sulfonate, sulfate, phosphonate, and phosphate groups.

Examples include: 4-[N,N-di(2-hydroxyethyl)-N-octadecylammonio]-butane-1-carboxylate; 5-[S-(3-hydroxypropyl)-S-hexadecylsulfonio]-3-hydroxypentane-1-sulfate; 3-[P,P-diethyl-P-3,6-trioxatetradecylophosphonio]-2-hydroxypropane-1-phosphate; 3-[N,N-dipropyl-N-3-dodecoxy-2-hydroxypropylam-

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monio]-propane-1-phosphonate; 3-(N,N-dimethyl-N-hexadecylammonio)propane-1-sulfonate; 3-(N,N-dimethyl-N-hexadecylammonio)-2-hydroxypropane-1-sulfonate; 4-[N,N-di(2-hydroxyethyl)-N-(2-hydroxydodecyl)ammonio]-butane-1-carboxylate; 3-[S-ethyl-S-(3-dodecoxy-2-hydroxypropyl)sulfonio]-propane-1-phosphate; 3-(P,P-dimethyl-P-dodecylphosphonio)-propane-1-phosphonate; and 5-[N,N-di(3-hydroxypropyl)-N-hexadecylammonio]-2-hydroxy-pentane-1-sulfate.

Examples of amphoteric surfactants which can be used in the compositions of the present invention are those which can be broadly described as derivatives of aliphatic secondary and tertiary amines in which the aliphatic radical can be straight chain or branched and wherein one of the aliphatic substituents contains from about 8 to about 18 carbon atoms and one contains an anionic water solubilizing group, e.g., carboxy, sulfate, sulfonate, phosphate, or phosphonate. Examples of compounds falling within this definition are sodium 3-dodecylaminopropionate, sodium 3-dodecylaminopropane sulfonate, N-alkyltaurines, such as the one prepared by reacting dodecylamine with sodium isethionate according to the teaching of U.S. Pat. No. 2,658,072, N-higher alkyl aspartic acids, such as those produced according to the teaching of U.S. Pat. No. 2,438,091, and the products sold under the trade name "Miranol" and described in U.S. Pat. No. 2,528,378. Other amphoteric surfactants such as betaines are also useful in the present composition.

Examples of betaines useful herein include the high alkyl betaines such as coco dimethyl carboxymethyl betaine, lauryl dimethyl carboxymethyl betaine, lauryl dimethyl alpha-carboxyethyl betaine, cetyl dimethyl carboxymethyl betaine, lauryl bis(2-hydroxyethyl)carboxymethyl betaine, stearyl bis(2-hydroxypropyl)carboxymethyl betaine, oleyl dimethyl gamma-carboxypropyl betaine, lauryl bis(2-hydroxypropyl) alpha-carboxyethyl betaine, etc. The sulfobetaines may be represented by coco dimethyl sulfoethyl betaine, stearyl dimethyl sulfoethyl betaine, lauryl bis(2-hydroxyethyl) sulfoethyl betaine, amido betaines amidosulfobetaines, and the like.

Many cationic surfactants are known to the art. By way of example, the following may be mentioned: stearyldimethylbenzyl ammonium chloride; dodecyltrimethylammonium chloride; nonylbenzylethyltrimethyl ammonium nitrate; tetradecylpyridinium bromide; laurylpyridinium chloride; cetylpyridinium chloride; laurylpyridinium chloride; laurylisoquinolinium bromide; diallow(hydrogenated)dimethyl ammonium chloride; dialkyltrimethyl ammonium chloride; and stearyltrimethyl ammonium chloride.

Many additional nonsoap surfactants are described in McCUTCHEON'S, DETERGENTS AND EMULSIFIERS, 1979 ANNUAL, published by Allured Publishing Corporation, which is incorporated here by reference.

The above-mentioned surfactants can be used in the liquid cleansing bath/shower compositions of the present invention. The anionic surfactants, particularly the alkyl sulfates, the ethoxylated alkyl sulfates and mixtures thereof are preferred. More preferred are C<sub>12</sub>-C<sub>14</sub> alkyl anionic surfactants selected from the group consisting of sodium alkyl glycerol ether sulfonate, sodium

lauroyl sarcosinate, sodium alkyl sulfate, sodium ethoxy (3) alkyl sulfate, and mixtures thereof.

Nonionic surfactants can be broadly defined as compounds produced by the condensation of alkylene oxide groups (hydrophilic in nature) with an organic hydrophobic compound, which may be aliphatic or alkyl aromatic in nature. Examples of preferred classes of nonionic surfactants are:

1. The polyethylene oxide condensates of alkyl phenols, e.g., the condensation products of alkyl phenols having an alkyl group containing from about 6 to 12 carbon atoms in either a straight chain or branched chain configuration, with ethylene oxide, the said ethylene oxide being present in amounts equal to 10 to 60 moles of ethylene oxide per mole of alkyl phenol. The alkyl substituent in such compounds may be derived from polymerized propylene, diisobutylene, octane, or nonane, for example.
2. Those derived from the condensation of ethylene oxide with the product resulting from the reaction of propylene oxide and ethylene diamine products which may be varied in composition depending upon the balance between the hydrophobic and hydrophilic elements which is desired. For example, compounds containing from about 40% to about 80% polyoxyethylene by weight and having a molecular weight of from about 5,000 to about 11,000 resulting from the reaction of ethylene oxide groups with a hydrophobic base constituted of the reaction product of ethylene diamine and excess propylene oxide, said base having a molecular weight of the order of 2,500 to 3,000, are satisfactory.
3. The condensation product of aliphatic alcohols having from 8 to 18 carbon atoms, in either straight chain or branched chain configuration with ethylene oxide, e.g., a coconut alcohol ethylene oxide condensate having from 10 to 30 moles of ethylene oxide per mole of coconut alcohol, the coconut alcohol fraction having from 10 to 14 carbon atoms. Other ethylene oxide condensation products are ethoxylated fatty acid esters of polyhydric alcohols (e.g., Tween 20-polyoxyethylene (20) sorbitan monolaurate).
4. Long chain tertiary amine oxides corresponding to the following general formula:



wherein R<sub>1</sub> contains an alkyl, alkenyl or monohydroxy alkyl radical of from about 8 to about 18 carbon atoms, from 0 to about 10 ethylene oxide moieties, and from 0 to 1 glyceryl moiety, and R<sub>2</sub> and R<sub>3</sub> contain from 1 to about 3 carbon atoms and from 0 to about hydroxy group, e.g., methyl, ethyl, propyl, hydroxy ethyl, or hydroxy propyl radicals. The arrow in the formula is a conventional representation of a semipolar bond. Examples of amine oxides suitable for use in this invention include dimethyldodecylamine oxide, oleyldi(2-hydroxyethyl) amine oxide, dimethyloctylamine oxide, dimethyldecylamine oxide, dimethyltetradecylamine oxide, 3,6,9-trioxaheptadecyldiethylamine oxide, di(2-hydroxyethyl)-tetradecylamine oxide, 2-dodecoxyethyldecylamine oxide, 3-dodecoxy-2-hydroxypropyldi(3-hydroxypropyl)-amine oxide, dimethylhexadecylamine oxide.

5. Long chain tertiary phosphine oxides corresponding to the following general formula:

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wherein R contains an alkyl, alkenyl or monohydroxy-alkyl radical ranging from 8 to 18 carbon atoms in chain length, from 0 to about 10 ethylene oxide moieties and from 0 to 1 glyceryl moiety and R' and R'' are each alkyl or monohydroxyalkyl groups containing from 1 to 3 carbon atoms. The arrow in the formula is a conventional representation of a semipolar bond. Examples of suitable phosphine oxides are: dodecyl dimethyl phosphine oxide, tetradecyl dimethyl phosphine oxide, 3,6,9-trioxaocetadecyl dimethyl phosphine oxide, cetyl dimethyl phosphine oxide, 3-dodecoxy-2-hydroxypropyl-di(2-hydroxyethyl) phosphine oxide, stearyl dimethyl phosphine oxide, cetyl ethylpropyl phosphine oxide, oleyl diethyl phosphine oxide, dodecyl diethyl phosphine oxide, tetradecyl diethyl phosphine oxide, dodecyl propyl phosphine oxide, dodecyl di(2-hydroxyethyl) phosphine oxide, dodecyl di(2-hydroxyethyl) phosphine oxide, tetradecyl methyl-2-hydroxypropyl phosphine oxide, oleyl dimethyl phosphine oxide, 2-hydroxydodecyl dimethyl phosphine oxide.

6. Long chain dialkyl sulfoxides containing one short chain alkyl or hydroxy alkyl radical of 1 to about 3 carbon atoms (usually methyl) and one long hydrophobic chain which contain alkyl, alkenyl, hydroxy alkyl, or keto alkyl radicals containing from about 8 to about 20 carbon atoms, from 0 to about 10 ethylene oxide moieties and from 0 to 1 glyceryl moiety. Examples include: octadecyl methyl sulfoxide, 2-undecyl methyl sulfoxide, 3,6,9-trioxaocetadecyl 2-hydroxyethyl sulfoxide, dodecyl methyl sulfoxide, oleyl 3-hydroxypropyl sulfoxide, tetradecyl methyl sulfoxide, 3-methoxytridecyl methyl sulfoxide, 3-hydroxytridecyl methyl sulfoxide, 3-hydroxy-4-decoxybutyl methyl sulfoxide.

The pH of the liquid cleansing bath/shower compositions herein is generally from about 8 to about 9.5, preferably from about 8.5 to about 9 as measured in a 10% aqueous solution at 25° C.

#### Method of Manufacture

The liquid soap cleansing compositions of the present invention may be made using techniques shown in the Examples. The preferred method for making the stable liquid comprises: (1) heating an aqueous (35-45% water) mixture of the soap/FFA to obtain a phase stable (liquid crystal) melt; (2) cooling the melt to room temperature to obtain a phase stable cream; and (3) diluting the cream with water to provide the stable dispersoidal liquid soap. These steps are preferably conducted under vacuum, but vacuum is not essential. Vacuum can be replaced with other deaeration methods, e.g., centrifugation. The dilution water preferably contains 0.5% PGE, 0.5% electrolyte, and 0.2% polymeric thickener to improve shelf stability. The preferred liquid soap has a shelf stable viscosity of from about 10,000 to about 80,000 cps (RVTDV-II, Spindle TD, 5 rpm). A viscosity of 30,000 cps ( $\pm 10,000$  cps) is ideal for dispensing this (high shear thinning) liquid from a standard piston-actuated displacement pump for personal cleansing. The preferred liquid soap can be formulated to be very mild by using a low soap concentration and selected higher saturated fatty acid soap chains. When a foam boosting surfactant, e.g., sarcosinate (2.5%), is added, the preferred liquid soap has very good lather.

The liquid soap cleansing compositions are useful as a cleansing aid for the entire body. The basic invention

may also be applicable in other liquid type products such as liquid hand soaps.

The following methods are used to evaluate liquid soap compositions:

#### Method I—Initial Viscosity (100% Product)

##### Apparatus:

Brookfield RVTDV-II Viscometer, Helipath, Spindle TD.

4 oz. Sample Jar

Conditions:

Sample Temperature Equilibrated to Room Temperature

(23° C./72°-77° F.), Brookfield at 5 rpm.

##### Method:

Transfer approximately 120 ml of product into 4 oz. sample jar taking care not to entrain air. Allow to equilibrate at room temperature for at least 4 hrs. Calibrate and zero viscometer referring to Brookfield manual. With TD spindle installed, viscometer at 5 rpm, and helipath stand energized (downward direction), lower viscometer until spindle is nearly touching product surface. Observe as helipath moves spindle through product surface and, as soon as spindle is submerged, begin timing. After 30 seconds record the next five viscosity readings. Average these readings and record. If the viscosity of the liquid soap is from about 10,000 to about 60,000 cps, it passes this test as a preferred liquid.

#### Method II—Viscosity Cycle (100% Product)

##### Apparatus:

Brookfield RVTDV-II Viscometer, Helipath, Spindle TD, 4 oz. Sample Jar, 120° F. (-49.5° C.) Constant Temperature Room or Water Bath.

##### Conditions:

Cycle sample from room temperature (RT) to 49.5° C. and return to room temperature. Sample residence time at 49.5° C. must be at least 8 hrs. and when returned to RT residence time must be at least 8 hrs. before viscosity is measured. Brookfield at 5 rpm.

##### Method:

Transfer approximately 120 ml of product into 4 oz. sample jar taking care not to entrain air. Place sample in constant temperature 49.5° C. room, oven or water bath. Maintain product at this temperature for at least 8 hrs. Transfer product to RT and allow to equilibrate for at least 8 hrs. Calibrate and zero viscometer referring to Brookfield manual. With TD spindle installed, viscometer at 5 rpm, and helipath stand energized (downward direction), lower viscometer until spindle is nearly touching product surface. Observe as helipath moves spindle through product surface and, as soon as spindle is submerged, time for 30 seconds and then record the next five viscosity readings. Average these readings and record. If the viscosity of the liquid soap is 10,000 to 60,000 cps, it passes this test for a more preferred liquid.

#### Method III—Accelerated Stability

##### Apparatus:

Centrifuge with temperature control capability or constant temperature room, 25-30 ml Flint Glass Vial.

##### Conditions:

Centrifuge samples at approximately 350g's and 120° F. (49.5° C.).

##### Method:

Transfer approximately 25 ml of product into glass vial taking care not to entrain air. Place sample in 49.5°

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C. atmosphere for at least 2 hrs. to equilibrate. Place vial into centrifuge with atmosphere controlled at 49.5° C. Centrifuge at approximately 350g's (350x force of gravity) 1200 rpm for 4 hrs. Remove from centrifuge and observe, note product separation, if any, and record result. If a liquid soap passes this test, it is highly preferred.

### EXAMPLES

The following examples further describe and demonstrate the preferred embodiments within the scope of the present invention. The Examples are given solely for the purpose of illustration and are not to be construed as limitations of the present invention as many variations thereof are possible without departing from its spirit and scope. Unless otherwise indicated, all percentages and ratios herein are approximations and by weight.

The following Example 1B is a preferred dispersoidal liquid soap of the present invention.

The Brookfield viscosity of 1B is about 30,000 cps. The Iodine Value of the fatty acids of Example 1 is about zero and its titer is about 59° C. Example 1B has totals of about 10.2% soap and 6.85% free fatty acid and 2.4% sarcosinate. The soap to free fatty acid (FFA) ratio is about 1:0.67.

TABLE 1

Formula Ingredients	EXAMPLE 1	
	1A Wt. %	1B Wt. %
Stearic Acid	7.55	4.53
Palmitic Acid	6.23	3.74
Myristic Acid	8.72	5.23
Lauric Acid	3.52	2.11
Triclosan	0.30	0.18
KOH (87%)	3.86	2.52
Glycerine	15.00	9.00
Mayquest (45%)*	0.44	0.26
Sodium Lauryl	13.33	8.00
Sarcosinate (30%)		
JR-400	0.50	0.30
Aloe Vera Powder	0.01	0.01
Perfume	0.30	0.18
Total Water (approx.)	50.00	70.00

\*Mayquest is a 50/50 mixture of HEDP/DPFA

A liquid soap (Example 1B) is made by first mixing the ingredients of "1A" as follows:

1. Mix and melt all of the fatty acids with the Triclosan into a jacketed vessel and heat to 80° C.
2. Dissolve the KOH pellets with water to make a 38% solution by weight.
3. Mix the glycerine, sodium lauryl sarcosinate, JR-400, Mayquest, and water in a separate jacketed vessel and heat to 80° C.
4. Transfer the melted fatty acid mix of Step 1 into a vacuum vessel which contains an internal homogenizer, wall scrapers and paddle mixers. E.g., a Mizuko Brand Automatic Driving Type Vacuum Emulsifier, Model APVQ-3DP, sold by Mizuko Industrial Co., Ltd., or a T.K. AGI Homo Mixer Model 2M-2, made by Tokushu Kika Kogyo Co., Ltd. While vacuum is not essential, it is highly preferred so that the intermediate product has a specific gravity of about  $\pm 1.05$ .
5. Slowly add the KOH solution under vacuum of about 400 mm Hg while mixing and homogenizing during saponifying. Maintain temperature controlled to 80 $\pm$ 5° C. while mixing.

6. After the saponification is complete, add the water mix of Step 3 under vacuum while continuing mixing and homogenizing. Maintain temperature controlled to 80 $\pm$ 5° C. while mixing to obtain a phase stable melt.
7. Immediately begin cooling from 80° C. to 50° C. at a 3° C./minute rate. Maintain mixing and vacuum during cooling step but stop homogenizing.
8. Dissolve the aloe vera powder in water and add at 50° C.
9. Cool from 50° C. to 35° C. at a 5° C./minute rate under vacuum and while mixing.
10. At 35° C. stop the vacuum and add the perfume. Continue cooling with mixing until final mix reaches about 30° C. At 30° C., stop cooling and unload the mix from the vessel.
11. The cooled melt of Step 10 (1A) is then diluted with distilled water at about room temperature. The water and the cooled melt is first mixed gently to provide a uniform slurry and then transferred to the vacuum vessel of Step 4 and homogenized for about 10 minutes under about 600 mm Hg to provide an aqueous (70% water) liquid soap dispersoidal (Example 1B). The liquid soaps can be made by varying the above method, but cannot be made by simple mixing of the ingredients of Example 1B.

TABLE 2

### EXAMPLES 2-6

Examples 2-6 are liquids made using the method of Example 1 except that the following stabilizing ingredients (finished liquid soap per cent) are added to the dilution water of Step 11:

TABLE 2

### EXAMPLES 2-6

Examples 2-6 are liquids made using the method of Example 1 except that the following stabilizing ingredients (finished liquid soap per cent) are added to the dilution water of Step 11:

KCl	0.5%
PGE	0.5%
Xanthan	0.2%

Examples 2-5 and Comparative Example 6

Ingredients	2		3		4		5		6	
	Wt. %		Wt. %		Wt. %		Wt. %		Wt. %	
Soap	10.2		5.0		5.0		20.0		20.0	
FFA	6.8		5.0		5.0		10.0		20.0	
Water	81.8		88.8		91.3		68.8		58.8	
Total	100.0		100.0		100.0		100.0		100.0	
Soap/FFA	1.0.66		1:1		1.0.5		1.0.5		1:1	

In short, Examples 2-6 are prepared in the following manner:

1. heating an aqueous (~50% water) mixture of the soap:FFA to obtain a phase stable melt (Step 6 above);
  2. cooling the melt to about room temperature; and
  3. diluting the cooled melt with water to provide a liquid soap.
- The dilution water of (3) contains the KCl, PGE and xanthan gum. The preferred liquid soap Example 2 has a Brookfield viscosity of 28,000 cps. Example 2 has a high shear thinning value and is ideal for dispensing from a standard piston actuated pump for personal cleansing. Example 2 is relatively mild due to its low soap concentration and higher chain saturated soap content. The IV is less than 1 and the titer is about 59.5 for the fatty matter used in Examples 2-6. The fatty matter of the liquid soaps used in Examples 2-6 are C<sub>12</sub>

at 13%±2%; C<sub>14</sub> at 35%±5%; C<sub>16</sub> at 24%±3%; and C<sub>18</sub> at 29%±3% on a total fatty matter basis.

Examples 2-5 are stable liquid dispersoids under normal conditions. Examples 4 and 5 separate under stress conditions defined hereinbelow as the Accelerated Stability Method III.

However, Examples 4 and 5 can be made more stable by increasing the levels of the stabilizing ingredients and/or by increasing the titer to over 60. Comparative Experimental Example 6 gels. Examples 2 and 3 are phase stable and shelf stable. Example 2 is preferred over Example 3 for better lather. The preferred liquid soap, e.g., Example 2, has a very rich creamy lather. However, in some of the following Examples, a foam-boosting surfactant, sarcosinate (2.4%), is added to enhance the rich and creamy lather.

In the following Examples 7-24, the ingredients shown as trade names are: Mayoquest is a 50/50 mixture of HEDP/DPTA. Triclosan is an antimicrobial. JR-400 is polyquaternium 10.

Capmul 8210 is mono/diglycerides of caprylic/capric acids (M.W. 250).

Caprol ET is mixed polyglycerol esters C<sub>12</sub>-C<sub>18</sub> (M.W. 2300).

Caprol 10G-4-0 is decaglycerol tetraoleate (M.W. 1800).

Acrysol ICS is polymeric thickener defined above.

TABLE 3

EXAMPLES 7 AND 8

Ingredients	7 Wt. %	8 Wt. %
Stearic Acid	4.53	4.53
Palmitic Acid	3.74	3.74
Myristic Acid	5.23	5.23
Lauric Acid	2.11	2.11
Triclosan	0.18	0.18
KOH (87%)	2.32	2.32
Glycerine	9.00	9.00
Mayoquest (45%)	0.26	0.26
Sodium Lauroyl Sarcosinate (30%)	8.00	8.00
JR-400	0.30	0.30
Aloe Vera Powder	0.01	0.01
Perfume	0.18	0.18
KCl	0.50	—
K-Acetate (55%)	—	1.20
Caprol ET	0.50	0.50
Caprol 10G-4-0	—	—
Capmul 8210	—	—
Acrysol ICS	—	—
Hydroxy Ethyl Cellulose (HEC)	—	—
Xanthan (M.W. 2,000,000)	0.20	0.20
D.I. Water	62.94	62.24
Accelerated Stability	Pass	Pass
Initial Viscosity	22,000	16,000
Cycle Viscosity	49,000	50,000

Examples 7 and 8 are two full liquid soap dispersoidal compositions with different electrolytes. Example 7, which is highly preferred, contains 0.5% KCl and 2.4% of the high lathering synthetic surfactant. Example 8 contains 1.20%×0.55 or 0.66% on an active basis of K-acetate. Both have acceptable viscosities. Example 7 is most preferred.

The total soap is 10.2% and the total FFA is 6.84%. The soap/FFA ratio is 1.0:0.67.

The level of electrolyte, K-acetate, is established as an equal molar concentration of KCl to the level of KCl used in Example 7.

The "Accelerated Stability" (Method III) is holding the liquid soaps at 120° F. (49.5° C.) for 4 hrs. under centrifuge (1200 rpm).

The "Viscosities" are measured at about 25° C. (RT) using a Brookfield RVTDV-II with Helipath Stand and a TD Spindle at 5 rpm.

TABLE 4

EXAMPLES 9-11

Ingredients	9 Wt. %	10 Wt. %	11 Wt. %
Stearic Acid	4.53	4.53	4.53
Palmitic Acid	3.74	3.74	3.74
Myristic Acid	5.23	5.23	5.23
Lauric Acid	2.11	2.11	2.11
Triclosan	0.18	0.18	0.18
KOH (87%)	2.32	2.32	2.32
Glycerine	9.00	9.00	9.00
Mayoquest	0.26	0.26	0.26
Sodium Lauroyl Sarcosinate (30%)	8.00	8.00	8.00
JR-400	0.30	0.30	0.30
Aloe Vera Powder	0.01	0.01	0.01
Perfume	0.18	0.18	0.18
KCl	0.50	—	—
K-Acetate (55%)	—	—	—
Caprol ET	—	—	—
Caprol 10G-4-0	—	—	—
Capmul 8210	0.50	—	—
Acrysol ICS	—	0.80	—
Hydroxy Ethyl Cellulose (HEC)	—	—	0.80
(M.W. 350,000-400,000)	—	—	—
Xanthan (M.W. 2,000,000)	0.20	—	—
D.I. Water	62.94	63.34	63.34
Accelerated Stability	Pass	Slight	Slight
Initial Viscosity	30,000	58,000	48,000
Cycle Viscosity	160,000	140,000	200,000

Example 9 contains 0.5% KCl; 0.50% Capmul 8210; and 0.20% xanthan. Examples 10 and 11 contain no KCl and, respectively, 0.80% Acrysol ICS and 0.80% HEC. The levels of water in these examples are slightly higher due to water added with KOH, sarcosinate, etc. Their initial viscosities are all acceptable for pumpable liquid soaps. The cycle viscosities are, however, too high. It failed the accelerated stability test, but is a stable dispersoidal liquid soap under normal conditions. Examples 10 and 11 separated only slightly under the accelerated stability test.

Compare Example 9 with Example 15 below. They are identical, but for the low molecular weight (250) nonionic Capmul 8210 in Example 9, which appears to have a negative effect on cycle viscosity stability. Example 12 (below) is also an identical formula. Its non-ionic is Caprol ET, which has a higher molecular weight (2300) than Capmul 8210. The higher molecular weight Caprol ET appears to have a positive effect on multiple cycle viscosities.

TABLE 5

EXAMPLES 12-15

Ingredients	12 Wt. %	13 Wt. %	14 Wt. %	15 Wt. %
Stearic Acid	4.53	4.53	4.53	4.53
Palmitic Acid	3.74	3.74	3.74	3.74
Myristic Acid	5.23	5.23	5.23	5.23
Lauric Acid	2.11	2.11	2.11	2.11
Triclosan	0.18	0.18	0.18	0.18
KOH (87%)	2.32	2.32	2.32	2.32
Glycerine	9.00	9.00	9.00	9.00
Mayoquest	0.26	0.26	0.26	0.26
Sodium Lauroyl Sarcosinate (30%)	8.00	8.00	8.00	8.00
JR-400	0.30	0.30	0.30	0.30



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TABLE 5-continued

Ingredients	EXAMPLES 12-15			
	12 Wt. %	13 Wt. %	14 Wt. %	15 Wt. %
Aloe Vera Powder	0.01	0.01	0.01	0.01
Perfume	0.18	0.18	0.18	0.18
KCl	0.50	—	0.50	0.50
K-Acetate (55%)	—	—	—	—
Caprol ET	0.50	0.50	0.50	—
Caprol 10G-4.0	—	—	—	—
Capmul 8210	—	—	—	—
Acrysol ICS	—	—	—	—
Hydroxy Ethyl Cellulose (HEC)	—	—	—	—
Xanthan	0.20	0.20	—	0.20
D.I. Water	62.94	63.44	63.14	63.44
Accelerated Stability	Pass	Pass	Pass	Pass
Initial Viscosity	22,000	42,000	46,000	24,000
Cycle Viscosity	49,000	185,000	37,000	40,000

Highly preferred Examples 12, 14 and 15 all have acceptable pumpable viscosities, initial and cycle, and pass the accelerated stability test. Examples 12, 14 and 15 have acceptable cycle viscosities and contain 0.5% KCl. Note that Example 13 does not contain an electrolyte cycle viscosity stabilizer and has an unacceptably high (185,000 cps) cycle viscosity. Example 14 contains no xanthan, but has an acceptable cycle viscosity. Caprol ET is a higher molecular weight (2300) nonionic and does not destroy the cycle viscosity in contrast to the lower molecular weight nonionic as used in Example 9.

TABLE 6

Ingredients	EXAMPLES 16-18		
	16 Wt. %	17 Wt. %	18 Wt. %
Stearic Acid	4.53	4.53	4.53
Palmitic Acid	3.74	3.74	3.74
Myristic Acid	5.23	5.23	5.23
Lauric Acid	2.11	2.11	2.11
Triclosan	0.18	0.18	0.18
KOH (87%)	2.32	2.32	2.32
Glycerine	9.00	9.00	9.00
Mayoquest	0.26	0.26	0.26
Sodium Lauroyl Sarcosinate (30%)	8.00	8.00	8.00
JR-400	0.30	0.30	0.30
Aloe Vera Powder	0.01	0.01	0.01
Perfume	0.18	0.18	0.18
KCl	0.50	—	—
K-Acetate (55%)	—	—	—
Caprol ET	—	—	0.50
Caprol 10G-4.0	—	—	—
Capmul 8210	—	—	—
Acrysol ICS	—	—	—
Hydroxy Ethyl Cellulose (HEC)	—	—	—
Xanthan	—	0.20	—
D.I. Water	63.64	63.94	63.64
Accelerated Stability	Pass	Fail	Fail
Initial Viscosity	37,000	11,000	24,000
Cycle Viscosity	35,000	222,000	180,000

Examples 16-18 all have acceptable initial viscosities. Example 16 has acceptable properties. Like Example 13, Examples 17 and 18 do not contain an electrolyte. Example 16 has 0.50% KCl and Examples 17 and 18 do not have the viscosity stabilizing electrolyte. Examples 17 and 18 also failed the accelerated stability test, but at room temp. are phase stable liquid soaps.

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TABLE 7

Ingredients	EXAMPLES 19-21		
	19 Wt. %	20 Wt. %	21 Wt. %
Stearic Acid	4.53	4.53	4.53
Palmitic Acid	3.74	3.74	3.74
Myristic Acid	5.23	5.23	5.23
Lauric Acid	2.11	2.11	2.11
Triclosan	0.18	0.18	0.18
KOH (87%)	2.32	2.32	2.32
Glycerine	9.00	9.00	9.00
Mayoquest	0.26	0.26	0.26
Sodium Lauroyl Sarcosinate (30%)	8.00	8.00	8.00
JR-400	0.30	0.30	0.30
Aloe Vera Powder	0.01	0.01	0.01
Perfume	0.18	0.18	0.18
KCl	0.50	—	0.50
K-Acetate (55%)	—	1.20	—
Caprol ET	0.50	0.50	0.50
Caprol 10G-4.0	—	—	—
Capmul 8210	—	—	—
Acrysol ICS	—	—	—
Hydroxy Ethyl Cellulose (HEC)	—	—	—
Xanthan	0.20	0.20	—
D.I. Water	62.94	62.24	63.14

Examples 19-21 are tested for multiple cycle viscosity stability. Their initial and multiple cycle viscosities are set out below in cps  $\times 1000$ .

	19	20	21
Initial	24	19	46
Cycle 1	44	50	37
Cycle 2	18	80-100	35-75
Cycle 3	26	60	28-45
Cycle 4	38	65	30-45
Cycle 5	35-60	—	—

TABLE 8

Ingredients	EXAMPLES 22-24		
	22 Wt. %	23 Wt. %	24 Wt. %
Stearic Acid	4.53	4.53	4.53
Palmitic Acid	3.74	3.74	3.74
Myristic Acid	5.23	5.23	5.23
Lauric Acid	2.11	2.11	2.11
Triclosan	0.18	0.18	0.18
KOH (87%)	2.32	2.32	2.32
Glycerine	9.00	9.00	9.00
Mayoquest	0.26	0.26	0.26
Sodium Lauroyl Sarcosinate (30%)	8.00	8.00	8.00
JR-400	0.30	0.30	0.30
Aloe Vera Powder	0.01	0.01	0.01
Perfume	0.18	0.18	0.18
KCl	0.50	0.50	0.50
K-Acetate (55%)	—	—	—
Caprol ET	—	—	—
Caprol 10G-4.0	—	—	0.50
Capmul 8210	—	—	—
Acrysol ICS	—	—	—
Hydroxy Ethyl Cellulose (HEC)	—	—	—
Xanthan	0.20	—	0.20
D.I. Water	63.44	63.64	62.94

The multiple cycle viscosities (cps  $\times 1000$ ) of Examples 22-24 are:

	22	23	24
Initial	24	6	N/A

-continued

	22	23	24
Cycle 1	40	43	N/A
Cycle 2	60-70	25-50	N/A
Cycle 3	60	45-75	N/A
Cycle 4	115	120-180	N/A
Cycle 5	—	75-130	N/A

N/A = not available.

The liquid cleansing composition preferably has an initial viscosity of from about 20,000 to about 40,000 cps and a cycle viscosity of from about 25,000 cps to about 70,000 cps.

A series of Examples are made to study the phase stability of the dispersoidal liquids. The levels of soap/fatty acid concentration is varied. See Table 9.

TABLE 9

EXAMPLES 25-28  
Soap Concentration Series  
(No Stabilizing Ingredients)

	25	26	27	28
Ingredients	Wt. %	Wt. %	Wt. %	Wt. %
% Soap	9.35	10.2	11.05	11.9
% FFA	6.27	6.84	7.41	7.98
Soap/FFA Ratio	1.0:67	1.0:67	1.0:67	1.0:67
Accelerated Stability	Fail	Fail	Fail	Fail
Initial Viscosity	23,000	38,000	50,000	55,000
Cycle Viscosity	110,000	145,000	155,000	155,000

Examples 25-28 without stabilizer are all room temp. phase stable liquid dispersoidal with acceptable initial viscosities; but all fail the accelerated stability test which is conducted under above stress conditions. See Method III above for details.

TABLE 10

EXAMPLES 29-31  
The effect of Fatty Acid Chain Length Distribution  
% Soap = 10.2  
% FFA = 6.84

These formulas also contained the stabilizing ingredients (0.2% Xanthan, 0.5% KCl, 0.5% PGE)

	29	30	31
Ingredients	Wt. %	Wt. %	Wt. %
% C <sub>12</sub> of Total FA's Mix	13.5	100	—
% C <sub>14</sub> of Total FA's Mix	33.5	—	—
% C <sub>16</sub> of Total FA's Mix	24	—	—
% C <sub>18</sub> of Total FA's Mix	29	—	100
Accelerated Stability	Pass	Pass	Pass
Initial Viscosity	28,000	15,200	4,000
Cycle Viscosity	79,200	740,000	17,200
Hand Lather	Good	Fair	Very Poor
Titer Point °C.	59.5	44.2	69.6

Examples 29-31 are formulated the same as Example 2, but for their fatty acid chains. The preferred soap chain mix is used in Example 29. They all pass the accelerated stability test. A mix containing some higher fatty acid chains and titers about 59.5° C. is preferred for cycle stability. Note that Examples 26 and 29 are the same but for the stabilizer in Ex. 29. The stabilizer appears to improve the accelerated stability and the cycle viscosity.

TABLE 11

EXAMPLES 32-34  
The Effect of Fatty Acid Chain Length Distribution  
% Soap = 10.2  
% FFA = 6.84

These formulas also contained the stabilizing ingredients (0.2% Xanthan, 5% KCl, 0.5% PGE)

	32	33	34
Ingredients	Wt. %	Wt. %	Wt. %
% C <sub>12</sub> of Total FA's Mix	50	62.5	12.5
% C <sub>14</sub> of Total FA's Mix	—	12.5	12.5
% C <sub>16</sub> of Total FA's Mix	—	12.5	12.5
% C <sub>18</sub> of Total FA's Mix	50	12.5	62.5
Accelerated Stability	Pass	Pass	Pass
Initial Viscosity	3,200	13,000	4,400
Cycle Viscosity	336,000	210,000	66,800
Hand Lather	Fair	Moderate	Poor
Titer Point °C.	56.9	50.9	63.7

Examples 32-34 are the same as Example 2, but for the soap chains. They all pass the accelerated stability test. The mixes with higher chains and titers of about 59.5° C. or above are preferred for cycle stability.

The initial viscosities of Examples 32 and 34 can be increased with the use of more thickener and salt in the formulation.

Referring to Table 12 below, three additional liquid soaps are made, but not shown, which have I. V.'s and titers of 11, 8, and 5 respectively. Their accelerated stabilities are good and their initial and cycle viscosities are 24,000 and 53,000, 5,200 and 60,800; and 3,200 and 36,000.

TABLE 12

EXAMPLES 35-38  
The Effect of Saturation  
% Soap = 10.2  
% FFA = 6.84  
Examples 35-38 also contain: 0.50% PGE, 0.5% KCl, and 0.2% Xanthan

	35	36	37	38
Ingredients	Wt. %	Wt. %	Wt. %	Wt. %
Iodine Value	<1.0	14	20	30
Accelerated Stability	Pass	Pass	Pass	Pass
Initial Viscosity	28,000	29,800	57,600	13,000
Cycle Viscosity	79,000	175,000	105,000	26,000
Hand Lather	Good	Very Poor	Very Poor	Poor

The most preferred Iodine Values are below 1 for stability and lather reasons. An additional benefit of low Iodine Values is no production of rancid odors due to the oxidation of the unsaturated double bond.

TABLE 13

EXAMPLES 39-41  
The Effect of Thickeners  
% Soap = 10.2  
% FFA = 6.84  
Soap/FFA Ratio = 1.0:67

	39	40	41
Ingredients	Wt. %	Wt. %	Wt. %
Thickener Type:	Acrysol	Hydroxy Ethyl Cellulose	Xanthan
Finished Product Level	0.80%	0.80%	0.20%
Accelerated Stability	Slight	Slight	Fail
Initial Viscosity	58,000	48,000	30,000
Cycle Viscosity	140,000	200,000	160,000

Table 13 supports:

(1) Thickeners improve the accelerated stability of the formula, compare with Example 26.

- (2) Thickeners by themselves (without electrolyte) do not appear to help the cycle viscosity stability.

TABLE 14

EXAMPLES 42-44  
The Effect of Nonionics (Polyglycerol Esters)

% Soap = 10.2

% FFA = 6.84

Soap/FFA Ratio = 1.0:67

Formulas also contained: 0.50% KCl and 0.2% Xanthan

Ingredients	42		43		44	
	Wt. %	Caprol ET	Wt. %	Caprol 10G-4-0	Wt. %	Capmul 8210
Nonionic Type	0.50%		0.50%		0.50%	
Finished Product						
Level						
Accelerated Stability	Pass		Pass		Pass	
Initial Viscosity	22,000		26,000		22,000	
Cycle Viscosity	49,000		31,000		260,000	

Caprol ET - mixed polyglycerol esters (HLB = 2.5, chain lengths C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, 6-10 glycerol units; M.W. = 2300).  
Capmul 10G-4-0 - decylglycerol tetrose (HLB = 6.2; M.W. = 1800).  
Capmul 8210 - mono/diglycerides of caprylic/capric acids (HLB = 5.5-6.0; M.W. = 230).

Table 14 supports: (1) Nonionics which have larger molecular weight (over about 1000) improve the cycle viscosity in the presence of electrolyte.

Example	Shear Thinning Factors		Shear Thinning Factor
	Viscosity (cps)	Viscosity (cps)	
	at 1 sec <sup>-1</sup>	at 10 sec <sup>-1</sup>	
1B	18,016	4,003	9.5
A	12,800	2,495	5
B	7,450	5,522	1.35
C	4,220	4,734	0.89
D	2,680	3,533	0.76

Examples A, B, C, and D are commercially available liquid personal cleansers, all packaged in pressure actuated pump containers. "A" is DOVE® Beauty Wash which claims to be a "non-soap" product. "B" is LIQ-UID IVORY® Soap, which is a K soap based product. "C" is Jergens Liquid and is a synthetic surfactant based product. "D" is Liquid Dial. Example 1B has a very high viscosity at a shear rate of 1 sec<sup>-1</sup>, but its high shear thinning factor (9.5) makes it possible to pump easily out of a pressure actuated pump. Examples B, C, and D have low shear thinning factors and, therefore, their viscosities are low to ensure pumpability.

Example 1B of the present invention is three times as viscous as DOVE® Beauty Wash and has a shear thinning factor about twice that of DOVE® Beauty Wash. A viscous product with a high shear factor is highly desirable for both pumpability and in use properties.

What is claimed is:

1. A dispersoidal liquid soap personal cleansing composition comprising:

- from about 5% to about 20% by weight of potassium fatty acid soap;
- from about 2.5% to about 18% C<sub>8</sub>-C<sub>22</sub> free fatty acid; wherein said fatty acid has an Iodine Value of from zero to about 15; and a titer of from about 44° to about 70° C.;
- from about 55% to about 90% water; and
- from about 0.1% to about 4% of a stabilizer selected from the group consisting of: from about 0.1% to about 2.0% of an electrolyte; and from 0% to about 2.0% of a polymeric thickener; and mixtures thereof; and

wherein said soap and said free fatty acid have a weight ratio of about 1:0.5 to about 1:1; and wherein said liquid has an initial viscosity of from about 4,000 cps to about 100,000 cps and a cycle viscosity of from about 10,000 cps to about 100,000 cps; and wherein said composition contains no more than 10% synthetic surfactant by weight of said composition.

2. A liquid cleansing composition of claim wherein said composition contains from about 0.3% to about 1% of said electrolyte which is selected from potassium chloride, potassium acetate and an equivalent molar concentration of any other water-soluble single charge electrolyte, and mixtures thereof; and from about 0.1% to about 1% of said thickener; and wherein said Iodine Value is less than 10 and said titer is from about 50 to about 70 and wherein said liquid has an initial 10,000 cps to about 60,000 cps and a cycle viscosity of from about 15,000 cps to about 80,000 cps.

3. A liquid cleansing composition of claim wherein said composition contains an electrolyte at a level of about 0.5% and is selected from potassium chloride, potassium acetate and an equivalent molar concentration of any other water-soluble single charge electrolyte, and mixtures thereof; and wherein said Iodine Value is less than 3 and said titer is from about 59 to about 70.

4. A liquid cleansing composition according to claim comprising from about 6% to about 14% by weight of said potassium soap and from about 4% to about 9% by weight of said free fatty acid; and wherein said liquid composition has an initial viscosity of from about 20,000 to about 40,000 cps and a cycle viscosity of from about 25,000 cps to about 70,000 cps.

5. A liquid cleansing composition according to claim 1 comprising from about 1% to about 10% of a high lathering synthetic surfactant.

6. A liquid cleansing composition according to claim 1 wherein the ratio of potassium soap to free fatty acid is from about 1:0.6 to about 1:0.8; and wherein said fatty acid is highly saturated and has an Iodine Value of from zero to about 10; and wherein said fatty acid is composed of alkyl chain lengths ranging from C<sub>8</sub> to C<sub>22</sub>; and wherein said fatty acid has a titer of from about 59 to about 70, and wherein said composition contains from about 2% to about 6% of a higher lathering synthetic surfactant.

7. A liquid cleansing composition according to claim 6 wherein said fatty acid has an Iodine Value of from zero to 3 and wherein said synthetic surfactant is sodium lauryl sarcosinate.

8. A liquid cleansing composition according to claim 1 wherein said composition has a shear thinning factor of at least 1.5 up to about 25.

9. A liquid cleansing composition according to claim 8 wherein said factor is from about 2 to about 20.

10. A liquid cleansing composition according to claim 8 wherein said shear thinning factor is from about 3 to about 15.

11. A liquid cleansing composition according to claim 1 wherein said fatty acid is composed of chain lengths ranging from C<sub>12</sub> to C<sub>18</sub>.

12. A liquid cleansing composition according to claim 1 wherein said composition contains from about 60% to about 80% water; from about 6% to about 14% said potassium fatty acid soap; from about 4% to about 9% said free fatty acid; and wherein said fatty acid has an Iodine Value of from zero to 3 and wherein said viscosity is from about 20,000 cps to about 40,000 cps.

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US006121214A

## United States Patent [19]

Exner et al.

[11] Patent Number: 6,121,214

[45] Date of Patent: Sep. 19, 2000

[54] USE OF THE ANTIBACTERIAL ACTIVE  
SUBSTANCE TRICLOCARBAN IN LIQUID  
SOAPS[58] Field of Search ..... 510/130, 131,  
510/137, 138, 155, 156, 159, 384, 386,  
414, 490, 101, 421, 501, 475[75] Inventors: Otto Exner, Ratingen; Manfred  
Hoffmann, Tönisvorst, both of  
Germany

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[21] Appl. No.: 09/381,423

[22] PCT Filed: Mar. 13, 1998

[86] PCT No.: PCT/EP98/01454

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§ 371 Date: Sep. 20, 1999

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§ 102(e) Date: Sep. 20, 1999

[87] PCT Pub. No.: WO98/42820

PCT Pub. Date: Oct. 1, 1998

[30] Foreign Application Priority Data

Mar. 25, 1997 [DE] Germany ..... 197 12 410

[51] Int. Cl.<sup>7</sup> ..... C11D 1/12; C11D 1/90;  
C11D 3/48[52] U.S. Cl. .... 510/130; 510/131; 510/137;  
510/138; 510/155; 510/156; 510/159; 510/384;  
510/386; 510/414; 510/490; 510/101; 510/421;  
510/501; 510/475

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[57]

## ABSTRACT

This invention relates to the use of triclocarban (N-(4-chlorophenyl)-N'-(3,4-dichlorophenyl) urea; trichlocarbanilide) as an antibacterial active ingredient in liquid soaps.

10 Claims, No Drawings

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# USE OF THE ANTIBACTERIAL ACTIVE SUBSTANCE TRICLOCARBAN IN LIQUID SOAPS

This application is a 371 of PCT/EP98/01454 filed Mar. 13, 1998.

## FIELD OF THE INVENTION

This invention relates to the use of triclocarban (N-(4-chlorophenyl)-N'-(3,4-dichlorophenyl)urea; triclocarbanilide) as an antibacterial active ingredient in liquid soaps.

## BACKGROUND OF THE INVENTION

Triclocarban has been used successfully as an antimicrobial active ingredient in antimicrobial bar soaps (solid soap) for almost 40 years. Triclocarban is only available in powdered form, e.g. crystalline (unground) or finely micronized. In contrast to other common antimicrobial active ingredients such as triclosan, chlorhexidine or chloroxylenol, which can be used both in antimicrobial bar soaps and in liquid soaps, triclocarban has hitherto not been incorporated into liquid soap.

The reason for this is the extremely low, inadequate solubility of triclocarban in liquid soaps and their main components. For example, the solubility of triclocarban in water, the main component of liquid soaps, is, at about 50-100 ppb, virtually zero. The solubility of triclocarban in common surfactants such as sodium lauryl sulphate or cocamidopropylbetaine and in common solvents such as glycerol or propylene glycol is well below 1%.

It is therefore problematic to incorporate triclocarban, given its activity profile and use concentrations known from bar soaps, into liquid soaps too.

Liquid soaps are here taken to mean washing products intended for hands and body, i.e. liquid soaps for hand and body, shower and/or bath gels, combined liquid shower and care products, such as shower gel having a body lotion function and the like.

The concentration of triclocarban in liquid soaps should preferably be from 0.3 to 0.5% for such products, although somewhat lower (0.1 to 0.25%) or somewhat higher (0.55 to 0.7%) concentrations can also be tried depending on the composition of the soap and microbiological testing.

Attempts have therefore been made to dissolve triclocarban in concentrations of 0.1 or 0.7% in commercially available liquid soaps as in Table 1 and to test for crystallization or undesired changes in appearance or chemical active ingredient instability over a few weeks at refrigeration temperature, room temperature and 40° C. (Preventol SB was used, min. 97% triclocarban).

TABLE 1

List of liquid soaps/shower and bath gels available on the German/European market

Shower and bath gel	Manufacturer/Supplier
CD ®	Elida Gibbs
Rexona ®	Elida Gibbs
Selina ®	Bayer AG
Azzurro paglieri ®	Schöhl
Pf (marie) ®	Heddat
Lux (Wash & Lotion)	Lever
Nivea (shower) ®	Beiersdorf
Ellen Betrix (Sport + Beauty) ®	Betrix
Chiff (ocean fresh) ®	Procter & Gamble
dusch das ®	Linger + Fisher

TABLE 1-continued

Liquid hand soap	Manufacturer
Seba MEBINACOR ®d ®	Sebapharma
Salina ®	Bayer AG
Lux ®	Lever
Kd ®	Thg-Vertriebs GmbH
Dove (cream wash) ®	Lever
Kur (cream soap) ®	win cosmetic/Aldi

List of liquid soaps available on the US market

Liquid soaps	Producer
Jergens liquid soap ®	The Andrew Jergens Company
Lever 2000 liquid soap ®	Lever Brothers Company
Clean & Smooth ®	Beckler Consumer Products Inc.

Triclocarban as powder cannot be dissolved in any of the 22 liquid soaps tested, not even with warming to about 50° C.

In a second test series, a 10% strength triclocarban presolution (triclocarban dissolved in a solubilizer/emulsifier of the PO/EO copolymer type) is incorporated into the above liquid soaps such that triclocarban is present in concentrations of 0.7%. Virtually all of the triclocarban crystallized out after just a few hours up to a maximum of 24 hours in 20 products. In the remaining 2 products, triclocarban also crystallized out, within a period of weeks. However, liquid soaps must have shelf lives of many months, meaning that these formulations too are unsuitable for triclocarban.

In contrast, triclosan can be incorporated into said liquid soaps in an amount of 0.7%.

The object of the invention was therefore to develop special, cosmetically exacting, ecologically and economically viable and microbiologically effective liquid soaps based on triclocarban which at the same time have a long shelf life.

Surprisingly, it has now been found that despite a high content of water and surfactants, cosmetically high-value and stable liquid soaps can be prepared.

## SUMMARY OF THE INVENTION

The application thus provides liquid soaps comprising

- |                               |                            |
|-------------------------------|----------------------------|
| a) 0.1 to 0.7% by weight of   | triclocarban and           |
| b) 11.2 to 14.7% by weight of | sodium lauryl sulphate and |
| c) 3 to 5% by weight of       | PEG-9 cocoglycerides or    |
| d) 2 to 10% by weight of      | PEG-9-glyceryl cocoate and |
| e) 0.2 to 0.4% by weight of   | PEG-400 (PEG-8) or         |
| f) 2.2 to 2.9% by weight of   | PEG-400 (PEG-12) and       |
|                               | Perfume and                |
|                               | Cocamidopropylbetaine and  |

at least one or more of the following components

- |                             |                                 |
|-----------------------------|---------------------------------|
| g) 1.0 to 2.5% by weight of | laureth 2 and/or laureth 3      |
| h) 0.1 to 0.2% by weight of | perservative                    |
| i) 0.7 to 1.0% by weight of | NaCl                            |
| j) 14 to 21% by weight of   | sorbitol                        |
| k) 0 to 2% by weight of     | polyisopate 20                  |
| m) 2.4% by weight of        | disodium laureth sulfosuccinate |

the difference to 100% by weight being made with water.

## DESCRIPTION OF THE INVENTION

Incorporation generally takes place by mixing triclocarban with the components of the liquid soap, or mixing

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triclocarban, optionally in the form of a presolution, with one or more individual components of the liquid soap to be prepared, and mixing the resulting mixture with the other remaining components, or mixing triclocarban in the form of a presolution in a solvent, which is suitable for dissolving and/or dispersing triclocarban and can be used in liquids, into the finished liquid soap afterwards.

It is also possible to mix individual components of the liquid soap or mixtures of individual components with triclocarban, which are then in turn optionally mixed with other individual components of the liquid soap or mixtures of individual components or solvents which likewise comprise triclocarban.

The application further provides that triclocarban is also incorporated in microencapsulated form into liquid washing products. This increases the solution behaviour and the stability of triclocarban to liquid washing products. Suitable microcapsules, e.g. those based on gelatine, have a diameter of a few  $\mu\text{m}$  to <1 mm and dissolve only during the washing operation, e.g. by rupturing.

Triclocarban is incorporated into the capsules in dissolved or disperse form, optionally with other components of the liquid detergent or with solvent. In principle, triclocarban can be used in liquid washing products (liquid soaps) combined with riclosan in any ratio in order to round off the antimicrobial or cosmetic profile of the liquid washing products.

Examples of novel liquid soaps are the following formulations:

#### EXAMPLES

Formulation 1	% by weight	% of active ingredient
sodium laureth sulphate	50	14
cocamidopropylbetaine	6	2.2
PEG-9 coeolyglycerides	4	4
PEG-400 (PEG 8)	10	10
triclocarban	0.1-0.5*	0.1-0.5
perfume	0.2-0.4	
laureth 2 (or laureth 3)	1.0	1.0
lauryl polyglycoses/ cocamidopropylbetaine	6	2.6
perservative	0.1-0.2	
NaCl	0.7	0.7
water	ad 100	about 65% total water content

\*At concentrations greater than 0.5%, triclocarban crystallizes out within weeks at room temperature.

Formulation 2	% by weight	% of active ingredient
sodium laureth sulphate	50	14
cocamidopropylbetaine	6	2.2
PEG-7 glyceryl cocoate	4.0	4.0
PEG 600 (PEG 12)	6.0	6.0
triclocarban	0.3-0.5*	0.3-0.5
perfume	0.2-0.4	
laureth 2 (or laureth 3)	1.0	1.0
lauryl polyglycoses/ cocamidopropylbetaine	6	2.6
perservative	0.1-0.2	

-continued

Formulation 2	% by weight	% of active ingredient
water	ad 100	about 70% total water content

\*At concentrations higher than 0.5%, triclocarban crystallizes out within weeks at room temperature.

Formulation 3	% by weight	% of active ingredient
sodium laureth sulphate	50	14
cocamidopropylbetaine	8.0	2.9
PEG-7 glyceryl cocoate	3.0	3.0
PEG 400 (PEG 8)	2.0	2.0
triclocarban	0.1-0.3*	0.1-0.3
perfume	0.2-0.4	4.2
perservative	0.1-0.2	
NaCl	0.7	0.7
water	ad 100	about 76% total water content

\*At concentrations higher than 0.3%, triclocarban crystallizes out within weeks at room temperature.

Formulation 4 preservative-free/self-preserving	% by weight	% of active ingredient
sodium laureth sulphate	52.5	14.7
cocamidopropylbetaine	7.5	2.7
PEG-9 coeolyglycerides	4.2	4.2
PEG-400 (PEG 8)	6.3	6.3
triclocarban	0.3-0.5*	0.3-0.5
perfume	0.2-0.4	
laureth 2 (or laureth 3)	1.6	1.6
erbitol	20.0-30.0	14.0-21.0
perservative	None	
water	ad 100	about 49-56% total water content

\*At concentrations higher than 0.5%, triclocarban crystallizes out within weeks at room temperature.

Formulation 5	% by weight	% of active ingredient
sodium laureth sulphate	40.0	11.2
cocamidopropylbetaine	8.0	2.9
disodium laureth sulphosuccinate	6.0	2.4
PEG 400 (PEG 8)	8.0	8.0
PEG-9 coeolyglycerides	5.0	5.0
polyorbate 20	0-2.0	0-2.0
triclocarban	0.3-0.5*	0.3-0.5
perfume	0.2-0.4	
laureth-2 (or laureth 3)	2.5	2.5
perservative	0.1-0.2	
NaCl	0.7-1.0	0.7-1.0
water	ad 100	about 62-64% total water content

\*At concentrations higher than 0.5%, triclocarban crystallizes out within weeks at room temperature.

Formulation 6	% by weight	% of active ingredient
sodium laureth sulphate	40.0	11.2
cocamidopropylbetaine	8.0	2.9
disodium laureth sulphosuccinate	6.0	2.4

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-continued

Formulation 6	% by weight	% of active ingredient
PEG 400 (PEG 8)	8.0	8.0
PEG-7 glyceryl cocoate	5.0*	5.0
polyisobutyl 20	0-2.0	0-2.0
tricloctan	0.3-0.7**	
perfume	0.2-0.4	
laureth-2 or (laureth 3)	2.5	2.5
preservative	0.1-0.2	
NaCl	0.7-1.0	0.7-1.0
water	ad 100	about 62-64% total water content

\*If PEG-9 cocoglyceride (see Formulation 5) is replaced by PEG-7 glyceryl cocoate, tricloctan can also be used in higher concentrations, such as, for example, here 0.7%.

\*\*At concentrations higher than 0.7%, tricloctan crystallizes out within weeks at room temperature.

Formulation 7	%	%
preservative-free/self-preserving		
sodium laureth sulphate	52.5	14.7
cocamidopropylbetaine	7.5	2.7
PEG-7 glyceryl cocoate	4.2*	4.2*
PEG 400 (PEG 8)	6.3	6.3
tricloctan	0.3-0.7**	0.3-0.7
Perfume	0.2-0.4	
laureth 2 (or laureth 3)	1.6	1.6
sorbitol	26.56	18.6
preservative	none	
water	ad 100	about 51% total water content

\*At concentrations higher than 0.7%, tricloctan crystallizes out in weeks at room temperature.

All of the above liquid soap formulations produce clear, colourless, viscous liquids with a pleasant odour. The pH is about 6-6.5. The viscosity can be adjusted to about 4000 mPas as desired, although lower-viscosity or higher-viscosity formulations are also possible without the stability of tricloctan being adversely affected.

Furthermore, in the Formulations 4 and 7, some of the water can be replaced by sorbitol. This gives liquid soaps which are so microbiologically stable that additional preservation is unnecessary. It is surprising that tricloctan, which is virtually insoluble in sorbitol, nevertheless remains stable in the liquid soap without crystallizing out.

The stability of the novel liquid soaps or of the crystallization behaviour of tricloctan and the formation of chloroanilines can be investigated as follows: Samples of all formulation liquid soaps containing 0.1-0.7% of tricloctan are stored at +6° C. (refrigerator), room temperature and at 40° C. (heating cabinet) for a period of at least 4 weeks. At intervals, checks are made to ascertain whether tricloctan has crystallized out.

In all of the above formulations, tricloctan remains in the given concentrations in solution and does not crystallize out. Concentrations higher than those given or significant modification of the formulations lead to the crystallization of tricloctan.

#### Example A

For the samples stored at room temperature and at 40° C., the possible formation of chloroanilines is investigated using HPLC. Tricloctan is specified as Preventol SB® (manufacturer: Bayer AG) having a maximum chloroanilines content of 450 ppm. From the use concentration, it is

possible to calculate a theoretical maximum starting chloroanilines content of 3.15 ppm. For the preparation of the liquid soaps, cold technology is used so that no chloroanilines can be formed during the preparation because there is no heat treatment or because the pH is not too high. The HPLC analysis method has a determination limit of about 10 ppm. As can be seen from Table 2, the chloroanilines content in the above formulations after storage for 11 or 14 weeks at room temperature is not crucial, being well below the FDA recommendation of 100 ppm. After storage for 4 or 11 weeks at 40° C. (accelerated stability test), a value no higher than 100 ppm of chloroanilines is likewise found.

TABLE 2

	Liquid soaps	Storage time	Total content of chloroanilines in ppm
15	Formulation 1	4 weeks, 40° C.	44
		14 weeks, RT	16
	Formulation 2	4 weeks, 40° C.	25
20		14 weeks, RT	18
	Formulation 3	6 weeks, 40° C.	15
		14 weeks, RT	nd
	Formulation 4	4 weeks, 40° C.	11
		11 weeks, RT	nd
25	Formulation 5	11 weeks, 40° C.	31
		11 weeks, RT	11
	Formulation 6	11 weeks, 40° C.	32
		11 weeks, RT	13
30	Formulation 7	4 weeks, RT	12
		11 weeks, RT	nd

nd =  $\leq 10$  ppm of chloroanilines

#### Example B

Microbiological investigation of the antibacterial action.

The test method used is a modified, quantitative suspension test in accordance with the test recommendations of the DGHM (Deutsche Gesellschaft für Hygiene und Mikrobiologie [German Association for Hygiene and Microbiology]). The test microbes used in the investigation are Gram-positive (resident skin flora) and Gram-negative (transient skin flora) bacteria. The microbial sowings (initial number of microbes) are  $8 \times 10^6$ - $1.1 \times 10^7$  CFU (colony-forming units) per ml of use solution, the liquid soap being used as a 75% strength dilution in deionized water (simulated use of liquid soap on wet hands). After a contact time of 1 minute, the reduction in number of microbes compared with the initial number of microbes is determined.

Result  
Reduction in the number of microbes in % in the liquid soap (75%) after a contact time of 1 minute, compared with the initial number of microbes.

Liquid soaps	Contact time in min	Test microbes Reduction in the number of microbes in %		
		<i>Staphylococcus aureus</i> ATCC 6538	<i>Lactobacillus plantarum</i> DSM 20205	<i>Pseudomonas aeruginosa</i> DSM 1117
		Gram-positive	Gram-positive	Gram-negative
Formulation 2	1	77-95	97.3	63-67.5
Formulation 3	1	91-97	50	55
Formulation 5	1	99	96	63

-continued

Liquid soaps	Contact time in min	Test microbes Reduction in the number of microbes in %		
		<i>Staphylococcus aureus</i> ATCC 6538	<i>Lactobacillus plantarum</i> DSM 20205	<i>Pseudomonas aeruginosa</i> DSM 1117
		Gram-positive	Gram-positive	Gram-negative
Formulation 6	1	99	91	92

To summarize, the formulations 1 to 7, for the given concentration ranges for triclocarban, are cosmetically modern, high-value, skin-friendly liquid soaps/shower and bath gels with high antimicrobial activity against gram-positive and gram-negative bacteria. These formulations are chemically-physically stable, the active ingredient triclocarban does not crystallize out, the formulations display fully the antimicrobial properties, and the content of possible decomposition products, chloroanilines, remains below the FDA recommended limit of 100 ppm.

Although the present invention has been described in detail with reference to certain preferred versions thereof, other variations are possible. Therefore, the spirit and scope of the appended claims should not be limited to the description of the versions contained therein.

What is claimed is:

1. A liquid soap comprising

a) 0.1 to 0.7% by weight of	triclocarban and
b) 11.2 to 14.7% by weight of	sodium laureth sulphate and
c) 3 to 5% by weight of	PEG-9 cocoglycerides or
d) 2 to 10% by weight of	PEG-400 (PEG-8) 2.0
e) 0.2 to 0.4% by weight of	PEG-600 (PEG-12) and
f) 2.2 to 2.9% by weight of	perfume and
	cocamidopropylbetaine and

at least one or more of the following components

g) 1.0 to 2.5% by weight of	laureth 2 and/or laureth 3 sulfate
h) 0.1 to 0.2% by weight of	perservative
i) 0.7 to 1.0% by weight of	NaCl
k) 14 to 21% by weight of	sorbitol
l) 0 to 2% by weight of	polyphosphate 20
m) 2.4% by weight of	disodium laureth sulfosuccinate

the difference to 100% by weight being made up by water.

2. The liquid soap of claim 1, wherein the soap comprises

sodium laureth sulphate	14
cocamidopropylbetaine	2.2
PEG-9 cocoglycerides	4
PEG 400 (PEG-8) 10	
triclocarban	0.3-0.5
laureth 2 or laureth 2 sulfate	1.0
lauryl polyglycoside	2.6
NaCl	0.7
wherein all amounts are expressed in percent by weight, the difference to 100% by weight being made up by water.	

3. The liquid soap of claim 1, wherein the soap comprises

sodium laureth sulphate	14
cocamidopropylbetaine	2.2
PEG-7 glyceryl cocoate	4.0
PEG 400 (PEG-12) 6.0	
triclocarban	0.3-0.5
laureth 2 or laureth 2 sulfate	1.0
lauryl polyglycoside	2.6
wherein all amounts are expressed in percent by weight, the difference to 100% by weight being made up by water.	

4. The liquid soap of claim 1, wherein the soap comprises

sodium laureth sulphate	14
cocamidopropylbetaine	2.9
PEG-7 glyceryl cocoate	3.0
PEG 400 (PEG-8) 2.0	
triclocarban	0.3-0.3
NaCl	0.7
wherein all amounts are expressed in percent by weight, the difference to 100% by weight being made up by water.	

5. The liquid soap of claim 1, wherein the soap comprises

sodium laureth sulphate	14.7
cocamidopropylbetaine	2.7
PEG-9 cocoglycerides	4.2
PEG 400 (PEG-8) 6.3	
triclocarban	0.3-0.5
laureth 2 or laureth 3 sulfate	14.7-14.0
sorbitol	
wherein all amounts are expressed in percent by weight, the difference to 100% by weight being made up by water.	

6. The liquid soap of claim 1, wherein the soap comprises

sodium laureth sulphate	11.2
cocamidopropylbetaine	2.9
disodium laureth	2.4
sulphosuccinate	
PEG 400 (PEG-8) 8.0	
PEG-9 cocoglycerides	5.0
polyphosphate 20	0-2.0
triclocarban	0.3-0.5
laureth 2 or laureth 3 sulfate	2.5
NaCl	0.7-1.0
wherein all amounts are expressed in percent by weight, the difference to 100% by weight being made up by water.	

7. The liquid soap of claim 1, wherein the soap comprises

sodium laureth sulphate	11.2
cocamidopropylbetaine	2.9
disodium laureth	2.4
sulphosuccinate	
PEG 400 (PEG-8) 8.0	
PEG-7 glyceryl cocoate	5.0
polyphosphate 20	0-2.0
laureth 2 or laureth 3 sulfate	2.5
NaCl	0.7-1.0



-continued

wherein all amounts are expressed in percent by weight, the difference to 100% by weight being made up by water.

8. The liquid soap of claim 1, wherein the soap comprises

sodium laureth sulphate	14.7
cocamidopropylbetaine	2.7
PEG-7 glyceryl cocoate	4.2
PEG 400 (PEG-8) 6.3	
triclocarban	0.3-0.7
laureth 2 or laureth 3 sulfate	1.6
sorbitol	18.6

wherein all amounts are expressed in percent by

-continued

weight, the difference to 100% by weight being made up by water.

9. Method for the preparation of the liquid soap of claim 1, wherein the triclocarban is mixed with the individual components of the liquid soap or triclocarban is mixed, optionally in the form of a presolution, with one or more individual components of the liquid soap to be prepared, and the resulting mixture is mixed with the other remaining components.

10. A method for making a liquid washing product comprising incorporating triclocarban in microencapsulated form into the liquid washing product of claim 1.

\* \* \* \* \*

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US006846785B2

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**(12) United States Patent**  
**Patel****(10) Patent No.: US 6,846,785 B2**  
**(45) Date of Patent: Jan. 25, 2005****(54) LIQUID SOAP WITH VITAMIN BEADS AND METHOD FOR MAKING SAME****(75) Inventor: Jayesh A. Patel, Chandler, AZ (US)****(73) Assignee: The Dial Corporation, Scottsdale, AZ (US)****(\*) Notice:** Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

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Primary Examiner—Necholus Ogden  
(74) Attorney, Agent, or Firm—Snell & Wilmer LLP**(57) ABSTRACT**

A liquid soap having vitamin-containing microcapsules including a base having an anionic surfactant and a chelating agent, a cross-linked acrylic polymer suspending agent, and multiple vitamin-containing microcapsules uniformly suspended in the liquid soap. The liquid soap is formulated at an elevated temperature which is maintained throughout the formulation process.

9 Claims, No Drawings

**(21) Appl. No.: 10/210,105****(22) Filed: Jul. 31, 2002****(65) Prior Publication Data**

US 2004/0023820 A1 Feb. 5, 2004

**(51) Int. Cl.<sup>7</sup> ..... A61K 7/00****(52) U.S. Cl. .... 510/130; 510/159; 510/425; 510/426; 510/429; 510/476; 510/477; 510/490****(58) Field of Search ..... 510/130, 159, 510/429, 425, 426, 476, 477, 490****(56) References Cited****U.S. PATENT DOCUMENTS**

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# LIQUID SOAP WITH VITAMIN BEADS AND METHOD FOR MAKING SAME

## FIELD OF INVENTION

The present invention generally relates to a liquid soap, and more particularly, to a liquid soap with vitamin beads or microcapsules and a method for making the same.

## BACKGROUND OF THE INVENTION

Liquid soaps and liquid soaps with vitamins are known in the art. Additionally, liquid personal cleansing products, including liquid soaps containing suspended beads or microcapsules, are also known in the art. Liquid soaps typically have a viscous liquid rheology, with low yield point, resulting in a liquid that is flowable even under conditions of low shear. In contrast, liquid soaps containing suspended beads or microcapsules typically have a gel-like rheology, with a pronounced yield point. This gel-like rheology is often required to prevent settling or other physical instability in the products during production, shipping, or use. However, the gel-like rheology is not a preferred rheology for a liquid soap or liquid cleansing product because of problems of product dispensing and aesthetics often associated with gel rheologies. Accordingly, there is a need met by the instant invention, for a liquid soap, or liquid personal cleansing compositions, capable of suspending beads or microcapsules in a stable fashion while simultaneously possessing a viscous liquid, as opposed to a gel-like, rheology.

Most liquid soap products are packaged in bottles having a pump type dispenser to facilitate use of the product without creating messy spills. However, the gel-type rheological make-up of the products often causes clogging of the pump due to collection of thick clumps of dried gel-type soap located on the outer opening of the pump dispenser.

The addition of vitamins and vitamin-containing beads to liquid soaps is also known in the art. However, because vitamin-containing beads or microcapsules in such products are generally suspended with xanthan gum, the products often appear hazy and have a more gel-like consistency rather than a viscous liquid consistency.

Accordingly, there is a need for a liquid soap having beads or microcapsules containing vitamins in which the beads or microcapsules are uniformly suspended in a stable fashion in the product, regardless of the temperature or viscosity of the liquid soap base in which the beads or microcapsules are suspended, and which said liquid soap deposits vitamins on the skin of the user during washing.

## SUMMARY OF THE INVENTION

In accordance with a preferred embodiment of the present invention, a liquid soap comprises a base having at least an anionic surfactant and a chelating agent, a plurality of microcapsules containing at least one vitamin, and a cross-linked acrylic polymer suspending agent, where the processing of the formulation of the liquid soap containing the microcapsules is performed at a temperature within a range of about 35 to 40 degrees C. A benefit of the composition is the deposition of vitamins to the skin surface while maintaining uniform suspension of the microcapsules at elevated temperatures and decreased breakage of the microcapsules at reduced temperatures. This composition has also been found to exhibit an appropriate amount of clarity with respect to its appearance and has been proven to be visually well accepted by consumers.

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The base preferably comprises a primary moisturizer, such as water, for example, which is mixed with an anionic surfactant such as, for example, ammonium lauryl sulfate and sodium laureth sulfate, and a chelating agent which may include, for example, but is not limited to tetrasodium EDTA or phosphates.

The microcapsules preferably comprise a natural polysaccharide matrix and active ingredients such as, for example, tocopheryl acetate and retinyl palmitate while the suspending agent is preferably a cross-linked, alkali-swellable acrylic emulsion polymer. Prior to formulation, the majority of the polymer's carboxyl functionality is in the protonated form. However, after neutralization of the polymer by adding it to the other ingredients of the liquid soap, its molecules ionize and expand to provide suspending and thickening properties. The microcapsules suspended in the liquid base preferably have a size within a range of about 700 to 1200 microns and preferably contain at least one of Vitamin A palmitate or Vitamin E acetate.

In accordance with a further aspect of the present invention, other ingredients may also be included in the liquid soap of the present invention. These include, for example, one or more humectants, one or more preservatives, one or more antibacterial agents, one or more fragrances, one or more nonionic surfactants, one or more antioxidants, one or more amphoteric surfactants, one or more colorants, and one or more neutralizers.

The present invention is also directed to a method for processing a liquid soap which includes the steps of preparing a liquid base, heating the liquid base to a temperature of about 35 to 40 degrees C. to form a batch liquid, maintaining the batch liquid at a temperature within a range of about 35 to 40 degrees C. throughout the entire formulation process, adding and mixing a plurality of microcapsules into the batch liquid; and adding and mixing a cross-linked acrylic polymer into the batch liquid. The step of preparing a liquid base may include adding at least one anionic surfactant and a chelating agent to a diluent such as water, for example. The method for processing the liquid soap may also include the steps of adding one or more of a humectant, a preservative, an antibacterial, a fragrance, a nonionic surfactant, an antioxidant, an amphoteric surfactant, a colorant, and a neutralizer. A second amphoteric surfactant may be added near the end of the process after addition of the cross-linked acrylic polymer to add viscosity. The batch liquid is continually mixed throughout the method for processing the liquid soap. Mixing methods are known in the art. A preferred mixing method is slow sweep mixing at a speed of 10 to 40 revolutions per minute (rpm) with side scrapers. Slow mixing with side scrapers and maintaining correct manufacturing temperature between 35 to 40 degrees C. can be utilized for any volume batch size of 1 pound to 100,000 pounds.

## DETAILED DESCRIPTION OF PREFERRED EXEMPLARY EMBODIMENTS

Preferred exemplary embodiments of the present invention will hereafter be described in conjunction with the description that follows. It will be understood that the detail provided herein is for illustration purposes only and that the subject invention is not so limited.

While the specific formulations of liquid soap within the present invention will be described in greater detail hereinafter, in general, a liquid soap formulation in accordance with the present invention comprises a liquid soap base, a plurality of microcapsules containing at least one

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skin benefit agent, e.g., a vitamin, and a suspending agent for suspending the microcapsules in the liquid soap base. Preferably, the microcapsules and suspending agent are added to the liquid base soap while temperature is maintained at a range of about 35 to 40 degrees C. Formulation of the liquid soap of the present invention at such elevated temperatures resulted in a formulation process which eliminated the introduction of air into the liquid soap product thereby enabling the production of a continuously clear looking product without air hubbles. Once the clear liquid soap product was obtained, care was taken to transport the product to bottles without the introduction of air into the product.

Preferably, the liquid base soap comprises an inert diluent or primary moisturizer, at least one anionic surfactant, and a chelating agent. The inert diluent or primary moisturizer preferably comprises water in an amount from about 20 to about 40 weight %, and more preferably from about 25 to about 35 weight %. Preferably, the anionic surfactant is present in a total amount of about 1 to 40 weight % and more preferably comprises ammonium lauryl sulfate in an amount of about 5 to 20 weight %, preferably 12 weight %, and sodium laureth sulfate, such as standapol ES-2 manufactured by Cognis, Inc., in an amount of about 1 to 40 weight %, and preferably 5 to 20 weight %. Specific surfactants that can be used in the base include, but are not limited to, lauryl sulfates, cetyl sulfates, 2-ethylhexyl sulfates, lauramine oxide, decyl sulfates, tridecyl sulfates, cocates, lauryl sarcosinates, lauryl sulfosuccinates, linear C<sub>10</sub> diphenyl oxide disulfonates, lauryl sulfosuccinates, lauryl ether sulfates (1 and 2 moles ethylene oxide), myristyl sulfates, olatees, stearates, tallates, cocamine oxide, decylamine oxide, myristamine oxide, ricinoleates, cetyl sulfates, and similar surfactants. Additional examples of surfactants can be found in "CTFA Cosmetic Ingredient Handbook," J. M. Nikitakis, ed., Washington, D.C. (1988) (hereafter CTFA Handbook), pages 10-13, 42-46, and 87-94, incorporated herein by reference. Suitable anionic surfactants include, but are not limited to, compounds in the classes known alkyl sulfates, alkyl ether sulfates, alkyl ether sulfonates, sulfate esters of an alkylphenoxy polyoxyethylene ethanol, alpha-olefin sulfonates, beta-alkoxy alkane sulfonates, alkylaryl sulfonates, alkyl monoglyceride sulfates, alkyl monoglyceride sulfonates, alkyl carbonates, alkyl ether carboxylates, fatty acids, sulfosuccinates, sarcosinates, octoxynol or nonoxynol phosphates, taurates, fatty laurides, fatty acid amide polyoxyethylene sulfates, isethionates, or mixtures thereof. Additional anionic surfactants are listed in McCutcheon's Emulsifiers and Detergents, 1993 Annuals, (hereafter McCutcheon's), McCutcheon Division, MC Publishing Co., Glen Rock, N.J., pp. 263-266, incorporated herein by reference.

The chelating agent is present in an amount of about 0.01 to 1.0 weight %, and more preferably about 0.02 to 0.05 weight %. Suitably, the chelating agent comprises tetrasodium EDTA or other EDTA salts.

The base liquid soap may include other additives such as humectants, preservatives, and vitamins. Humectants may comprise about 0.1 to 5 weight % of the base liquid while preservatives may comprise about 0.1 to 1.0 weight % of the base liquid. Suitable humectants include glycerin and any other humectants listed in the CTFA Handbook, which are herein incorporated by reference, and suitable preservatives include DMDM hydantoin and any other preservatives listed in the CTFA Handbook, which are also herein incorporated by reference. Vitamins, such as Vitamin E acetate and

Vitamin A palmitate, for example, may be present in the base liquid soap in an amount of about 0.1 to 1.0 weight %, and preferably in an amount of about 0.01 to 0.5 weight %.

The liquid soap of the present invention may also include other conventional additives such as antibacterials, nonionic surfactants, antioxidants, amphoteric surfactants, neutralizers, colorants, and fragrances. The amounts of such additives will be dependent upon the desired liquid soap end product of the present invention. The final liquid soap product of the present invention may generally comprise antibacterials in an amount of about 0.1 to about 1.0 weight %, nonionic surfactants in an amount of about 1 to about 40 weight %, amphoteric surfactants in an amount of about 1 to 10 weight %, and neutralizers in an effective amount for neutralization of the acrylates polymer. Consumer acceptable amounts of dyes and fragrances may also be added in sufficient amounts to perform their intended function without adversely affecting the clarity and stability of the base liquid soap. All such additives are added to the base liquid soap while maintaining a product temperature between about 35 to 40 degrees C.

The liquid soap of the present invention also includes microcapsules or beads which contain at least one vitamin, such as Vitamin E and/or Vitamin A. The microcapsules preferably comprise a natural polysaccharide matrix such as, for example, agar/alginate/chitosan, which contains active ingredients such as tocopheryl acetate and retinyl palmitate. The tocopheryl acetate and the retinyl palmitate preferably comprise about 0.1 weight % and about 0.01 weight %, respectively, of the microcapsules. The microcapsules containing vitamins preferably have a size of about 700 to 1200 microns and are preferably contrasting color in order to maximize contrast with the base liquid soap. An example of one preferred bead includes a bead having mica to make the beads shiny and further enhance their visual effect. The microcapsule containing vitamins are preferably custom designed by a manufacturer such as Cognis Iberia S.L. located in Spain which can make the core of the microcapsules hard enough to withstand the surfactants contained in the liquid soap composition while also manipulating the internal phases of the microcapsules with mineral oil and glycolic acid in order to have the microcapsules achieve densities that are similar to the density of the liquid soap so that the microcapsules can be uniformly suspended in the liquid soap also suspended without settling or creaming.

The microcapsules are an essential part of the liquid soap product of the present invention in that they add both functionality and an attractive appearance to the liquid soap product. The microcapsules must have a hard external face to withstand the chemical action of the surfactant system contained within the liquid soap and must also withstand physical manipulation and mechanical breakage. The core and internal phase of the microcapsules must also be able to withstand harsh temperature changes because processing and transportation may expose the product to extremes of temperature.

The liquid soap of the present invention also includes a cross-linked acrylic polymer which functions as a suspending agent for the vitamin-containing microcapsules. The cross-linked acrylic polymer is preferably an alkali-swellable acrylic emulsion polymer having the majority of its carboxyl functionality in the protonated form. Prior to neutralization, the polymer molecules are coiled and impart relatively little suspension and viscosity. Once neutralized, the molecules ionize and expand due to the charge repulsion of the anionic carboxylate and thereby provide suspending and thickening properties. Preferably, the cross-linked

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acrylic polymer is present in an amount of about 5.75 to 7.00 weight % of the final liquid soap product of the present invention and preferably comprises Carbopol AQUA SF-1 polymer manufactured by BF Goodrich. Carbopol AQUA SF-1 which is approximately 70 weight % water and 30 weight % proprietary polymer/solids. It is recommended that Carbopol AQUA SF-1 be added to the free water of a formulation with gentle mixing at the start of the batching process. It is also recommended that amphoteric surfactants be added prior to neutralizing the Carbopol AQUA SF-1.

However, in contrast to the Carbopol AQUA SF-1 recommendations, the liquid soap of the present invention is formulated by adding diluted carbopol AQUA SF-1 near the end of the formulation process after adding surfactants. In addition, with the liquid soap of the present invention, an amphoteric surfactant may also be added after neutralizing the Carbopol AQUA SF-1.

In formulating the liquid soap of the present invention, the base liquid soap of the present invention is first formulated by adding at least one anionic surfactant and a chelating agent to an inert diluent, such as water, and heating the solution to a temperature of about 35 to 40 degrees C., and preferably about 36 to 37 degrees C. Other ingredients may also be added to the base liquid soap such as humectants and preservatives. Mixing of the base liquid soap should be maintained at about 10 to 40 revolutions per minute throughout the batch and the temperature should be maintained between 35 to 40 degrees C. throughout the batch. Once all ingredients are added to the base liquid soap, the batch should be mixed for approximately 5 minutes or until the mixture is clear.

Next, several additives may be incorporated into the mixture prior to the addition of the vitamin-containing microcapsules. Antibacterials, fragrance, nonionic surfactants, and antioxidants may each be added one at a time to the batch and mixed thoroughly between additions. After all are added, the batch is preferably mixed for about 15 minutes while the temperature continues to be maintained between about 35 to 40 degrees C. An amphoteric surfactant may then be added to the batch and mixed into the batch for approximately 10 minutes while continuing to maintain the temperature between about 35 to 40 degrees C.

Vitamin-containing microcapsules such as those described above are then added to the batch and mixed into the batch. The mixing speed is adjusted to adequately disperse the microcapsules into the batch while still maintaining the temperature between about 35 to 40 degrees C. One or more colorants are then added to the batch and mixed into the batch. The colorants may also be added to the batch prior to adding the vitamin-containing microcapsules.

The cross-linked acrylic polymer, such as Carbopol AQUA SF-1, is then premixed with an inert diluent, such as water, in a ratio of preferably about 3 to 1, or a little less, and is then slowly added to the batch while maintaining the temperature of the batch between about 35 to 40 degrees C. The batch is then mixed for at least 10 minutes after adding the diluted cross-linked acrylic polymer. A neutralizer, such as sodium hydroxide pellets predissolved in warm water, is then slowly added to the batch until the batch clears while maintaining the 35 to 40 degree C. temperature. Sodium hydroxide is added in an amount sufficient to achieve a preferable pH of about 5 to 7. To achieve clarity, the batch will require mixing the batch for about 10 to 15 minutes while maintaining the temperature of the batch. Finally, an amphoteric surfactant such as cocamidopropyl betaine and cocamide MEA manufactured under the trade name Mack-

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man BC 39 by McIntyre, Inc., based in Chicago, in an amount of about 1 to 10 weight % of the final liquid soap product is added to the batch to add viscosity and is mixed until clear while maintaining the batch temperature. In one preferred method for making the liquid soap with vitamin beads of the present invention, the batch containing the final liquid soap product is mixed for about 15 minutes after obtaining a clear product.

A preferred final composition in accordance with the present invention includes the following components, all of which are listed in weight percent of the final product:

## EXAMPLE 1

Ingredient	Weight %
Purified water	32.286
Ammonium lauryl sulfate	12.000
Sodium laureth sulfate	18.000
Glycerin	1.000
Tetra sodium EDTA	0.050
DMDM hydantoin	0.400
DL Panthenol	0.020
Triclosan	0.150
Fragrance	0.300
Peg-18 glyceryl oleate/cocotate	0.880
Decyl polyglucose	3.330
Tocopheryl acetate	0.200
Cocamidopropyl betaine	1.820
Vitamin-containing microcapsules	0.400
0.1% soln. of FD&C yellow No. 6	0.081
0.1% soln. of FD&C red No. 4	0.023
Water	17.000
Acrylates copolymer	6.200
Water	2.000
Sodium hydroxide	0.180
Cocamidopropyl betaine and cocamide MEA	3.680
TOTAL	100.000

It should be appreciated that the above formulation is set forth in an illustrative manner and that other liquid soap formulations having similar compositions and formulation steps are within the scope of the present invention. For example, other specific formulations which achieve near similar results with respect to clarity, uniform suspension of beads, vitamin deposition, and stability include:

## EXAMPLE 2

Ingredient	Weight %
Purified water	32.736
Ammonium lauryl sulfate	12.000
Sodium laureth sulfate	18.000
Glycerin	1.000
Tetra sodium EDTA	0.050
DMDM hydantoin	0.400
DL Panthenol	0.020
Triclosan	0.150
Fragrance	0.300
Peg-18 glyceryl oleate/cocotate	0.880
Decyl polyglucose	3.330
Tocopheryl acetate	0.200
Cocamidopropyl betaine	1.820
Vitamin-containing microcapsules	0.400
0.1% soln. of FD&C yellow No. 6	0.081
0.1% soln. of FD&C red No. 4	0.023
Water	17.000
Acrylates copolymer	5.750

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Ingredient	Weight %
Water	2.000
Sodium hydroxide	0.180
Cocamidopropyl betaine and cocamide MEA	3.680
TOTAL	100.000

## EXAMPLE 3

Ingredient	Weight %
Purified water	32.486
Ammonium lauryl sulfate	12.000
Sodium laureth sulfate	18.000
Glycerin	1.000
Tetra sodium EDTA	0.050
DMDM hydantoin	0.400
DL Panthoel	0.020
Triclosan	0.150
Fragrance	0.300
Peg-18 glyceryl oleate/cocate	0.880
Decyl polyglucose	3.330
Tocopheryl acetate	0.200
Cocamidopropyl betaine	1.820
Vitamin-containing microcapsules	0.400
0.1% soln. of FD&C yellow No. 6	0.081
0.1% soln. of FD&C red No. 4	0.023
Water	17.000
Acrylates copolymer	6.000
Water	2.000
Sodium hydroxide	0.180
Cocamidopropyl betaine and cocamide MEA	3.680
TOTAL	100.000

## EXAMPLE 4

Ingredient	Weight %
Purified water	29.893
Ammonium lauryl sulfate	12.000
Sodium laureth sulfate	18.000
Glycerin	1.000
Tetra sodium EDTA	0.050
DMDM hydantoin	0.400
DL Panthoel	0.000
Triclosan	0.150
Fragrance	0.250
Peg-18 glyceryl oleate/cocate	0.880
Decyl polyglucose	3.330
Tocopheryl acetate	0.250
Cocamidopropyl betaine	1.820
Vitamin-containing microcapsules	2.000
FD&C yellow No. 6	0.00619
FD&C red No. 4	0.00033
Water	17.000
Acrylates copolymer	7.000
Water	2.000
Sodium hydroxide	0.250
Cocamidopropyl betaine and cocamide MEA	3.680
TOTAL	100.000

The following materials were used as ingredients in the examples:

a) Alkyl polyglucoside (APG) manufactured by Henkel Co. in Hoboken, N.J. having the trade name PLANTAREN 2000 was used for the decyl polyglucose,

b) Sodium Lauryl ether sulfate, 2 mole EO (SLES-2), manufactured by Henkel Corp. having the trade name STANDAPOL-ES-2 was used as the sodium laureth sulfate,

c) Triclosan (TCS), IRGASAN DP-300 manufactured by Ciba Specialty Chemical Corp. in Greensboro, N.C. was used for the triclosan,

d) Glycerin (GLY) manufactured by Henkel/Emery in Cincinnati, Ohio was used for the glycerin, and

e) Carbopol AQUA SF-1 polymer manufactured by B.F. Goodrich was used for the acrylates copolymer.

Examples of materials used for other listed ingredients can be found in U.S. Pat. No. 6,107,261 which is herein incorporated by reference in its entirety. The antibacterial composition described in U.S. Pat. No. 6,107,261 may comprise part of the base liquid soap of the present invention with some manipulation depending on the amounts of other ingredients listed above that are added to the liquid soap of the present invention.

Typically, most oils such as Vitamin E Acetate and Vitamin A Palmitate will wash off in surfactant based products. However, the formulation of the liquid soap of the present invention includes the steps of premixing the microcapsules containing the Vitamin E Acetate and/or Vitamin A Palmitate. This allows the Vitamin E Acetate and/or Vitamin A Palmitate to solubilize, aiding deposition of the vitamins on the skin of the user, and also prevents the product from becoming hazy in appearance.

The liquid soap of the present invention has many advantages. The liquid soap product of the present invention comprises a clear base with contrasting uniformly suspended vitamin-containing capsules. The liquid soap of the present invention contains a minimum number of air bubbles which completely disperse over time thereby enhancing the clarity of the product. The liquid soap of the present invention also enables deposition of vitamins contained in the formula to the skin surface.

Formulating the liquid soap of the present invention at a temperature of about 35 to 40 degrees C. both reduced air entrapment, thereby avoiding air bubbles, and increased the clarity of the product. The suspending agent used in the liquid soap of the present invention enables uniform suspension of the microcapsules regardless of the temperature and viscosity of the base liquid soap containing surfactants and is also effective in helping to produce a clear product. The liquid soap of the present invention has a liquid rheology, unlike the gelled rheology of other bead containing soaps, and preferably has a viscosity of about 5000 to 15000 centipoise using a Brookfield viscometer model LVF spindle #3 at 12 rpm and at 25 degrees C. The viscous liquid of the present invention, unlike gels, achieves a low yield stress.

The above described examples were tested for stability at one, two, three, four, eight and twelve weeks post production and were found to exhibit stable and acceptable pH and viscosity values. Freeze-thaw stability results of the product were also favorable.

It will be understood that the foregoing description is of preferred exemplary embodiments of the present invention, and that the present invention is not limited to the specific examples and compositions set forth herein. Such examples and compositions are for illustrative purposes only. Various modifications may be made in light thereof as will be suggested to persons skilled in the art without departing from the scope of the invention as expressed in the appended claims.

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What is claimed is:

1. A liquid soap comprising:

about 65 to about 75 weight % of a liquid base;

about 0.1 to about 2.1 weight % of microcapsules; and

about 20 to about 25 weight % of a suspending agent  
comprising a cross-linked acrylic polymer and water;  
wherein during formulation the liquid base is maintained at  
a temperature between about 35 to 40 degrees C. during  
addition of all other ingredients including the microcapsules  
and the suspending agent.

2. The liquid soap of claim 1 wherein the liquid base  
comprises an anionic surfactant and a chelating agent.

3. The liquid soap of claim 2 wherein the liquid base  
further comprises at least one of a humectant, a preservative,  
and a vitamin.

4. The liquid soap of claim 1 further comprising at least  
one of an antibacterial, a fragrance, a nonionic surfactant, an  
antioxidant, an amphoteric surfactant, a colorant, and a  
neutralizer.

5. The liquid soap of claim 1 wherein the microcapsules  
contain at least one of Vitamin A and Vitamin E.

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6. The liquid soap of claim 1 wherein said microcapsules  
have a size within a range of about 700 to 1200 microns.

7. The liquid soap of claim 1 wherein the suspending  
agent comprises an alkali-swellable acrylic emulsion poly-  
mer.

8. The liquid soap of claim 3 wherein the liquid base  
comprises:

about 20 to 40 weight % of a diluent;

about 1 to 40 weight % of an anionic surfactant;

about 0.1 to 5 weight % of a humectant;

about 0.01 to 1.0 weight % of a chelating agent;

about 0.1 to 1.0 weight % of a preservative; and

about 0.1 to 1.0 weight % of a vitamin.

9. The liquid soap of claim 4 wherein said amphoteric  
surfactant comprises about 1.0 to 10.0 weight % of said  
liquid soap.

\* \* \* \* \*

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FROM: <http://home.earthlink.net/~skinesscentuals/Liquid.html>



## liquid soap ...from scratch

return to  
front page

you would think that once you become comfortable with making soap from scratch, it would be an easy segue to making liquid soap. *au contraire!* as covered in "bar soap from scratch", cp (cold process) soapmaking is easy, (which accounts for it's popularity), as compared to hp (hot process) soapmaking. it's not so much that hp is more difficult, just more involved. plus, the "hot process" or "cooking" makes the soap itself harder to work with. although liquid soap falls into two categories: "bar-soap conversion" and "hot process", the latter is the most involved and is necessary to produce a viscous, transparent, clear liquid soap.

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i first went the easy route, reviewing recipes for a simple way of converting solid soap to liquid form. although the procedures varied, all the recipes basically came down to grating bar soap and diluting it with water. if this dilution is too thin, it's not effective a soap ...it doesn't lather [well]. if it's too thick, it wants to congeal into an "unusable" glob of slimy goo. although I came out with a usable product by following this recipe for "bar-soap conversion", this was not what i wanted.

since the results of converting bar soap to a liquid proved unsatisfactory, i decided to tackle the hot process method for making a transparent liquid soap from scratch. the first step was to get [one of] catherine failor's books\* entitled "*making natural liquid soap*". i did, and as i read, i realized that this was a bit more involved. but just as i overcame my initial apprehension of cp soapmaking, i was sure i could conquer this. after perusing the manual several times and sifting through all the facts and details, i proceeded with the method outlined below.



click to enlarge

(\*subsequently, i obtained ms. failor's other soapmaking publications entitled "*making cream soap*" and "*making transparent soap*". i've chronicled my exploits working with these fascinating and very-different soaps on my "cream soap" and "transparent soap" pages.). and lastly, there's "whipped soap", which i've added to round out the soapmaking repertoire.

this is the method i used for the hot process liquid soap: -- (see "soap recipes" for my liquid soap recipes)

\* \* \*



## 12 determining the concentration.

*the concentration of the soap is not just a matter of choice. it's also determined by the oils you used. a concentration that's too low produces a soap that won't lather effectively, too high and the soap will congeal back into a paste.*

*soap from softer oils can only withstand so much concentration before it starts to congeal; sometimes as little as 20%. soap from all hard oils (e.g. coconut) can take a concentration of up to 40%. soap made from a mixture of hard and soft oils takes a concentration from 25% to 35%.*

*among other things, borax is an emulsifier and adding it to your soap will allow the soap to sustain a [higher] concentration where it would otherwise start to congeal. i'm going for a concentration of 30%.*

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<u>soap</u> <u>concentration</u>	<u>water</u> <u>per</u> <u>lb. of</u> <u>paste</u>
15 percent	48 ounces
20 percent	32 ounces
25 percent	22 ounces
30 percent	16 ounces
35 percent	12 ounces
40 percent	9 ounces

*use the table to the left or calculate for yourself; the formula for determining dilution is simple. find out the percentage of actual soap in your paste (divide weight of (lye and oils) by the weight of the paste. this will be around 64% - 66%. now multiply the weight of the paste by that percentage. this is the weight of the actual soap in the paste. now, divide that amount by the desired concentration, (say 30%). this is the projected weight of your dilution. finally, subtract the weight of the paste from this projected dilution weight. the result is the amount of water you need to add to the paste to achieve the selected concentration.*

*for 100 gm pf paste with a 65% of actual soap, diluted for a 30% concentration, the numbers are as follows: (100 times .65 = 65; divided by .30 = 216.6; minus 100 = 116.6 gm water needed).*